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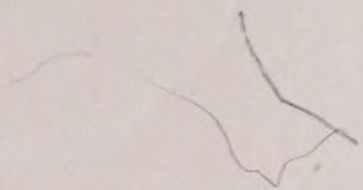

A PRACTICAL COURSE

IN THE

Fire Assaying
for
Gold, Silver and Lead
of
Ores, Bullion and Metallurgical Products

BY

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AUTHOR'S EDITION, 1900

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General Directions and Suggestions.

You should read carefully the directions for what you are about to do before commencing work each day, even if you have read them before, in order to refresh your memory. You should guard against losing or mislaying your assaying note-book, either while working through the course or afterwards, for the reasons that, as the work goes on, it will come to be an original record, of *your own* methods and results in assaying, by which you will judge the methods given in books; and if lost, it cannot be duplicated, so far as your own notes and results are concerned.

THE KEEPING OF NOTES.

It will be quite easy to give the notes a neat appearance and to make them readily legible and permanent if you use for writing them only either a fountain pen or a drawing pencil. It is well to place the date at the beginning of the note-book record for each day. A neat report or notes not only creates a good first impression but is often of value as an index of care and correctness in other more important matters; while an unsightly slovenliness in the record or report is apt to produce an unfavorable impression at the first glance and create distrust.

Numerical results can almost always be more briefly and clearly stated in tabular form. Forms for this purpose will be given from time to time; but you are encouraged to devise neat forms for your own use. Numerical results should be written in at once in the proper place in permanent form as obtained at the balance or the burette; and calculations should be worked out at once or at your earliest convenience.

Not all which you should observe and record, however, can be best put in tabular form. In your work in the laboratory, you should not be satisfied to carry through in a mechanical manner the necessary operations; but should strive to acquire an intimate knowledge of the phenomena taking place by exercising and still farther cultivating powers of accurate and keen observation and by adducing, so for as you can, the chemical or physical explanation of the phenomena seen. The possession of broad and intimate knowledge of details, learned first-hand from working with the things themselves, the details so learned being co-ordinated and arranged by reflection and,

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where necessary, accompanied by a corresponding skill of hand, is what distinguishes any man worthy of the name of expert in any kind of work. Such power of observation is worth its acquisition for use in assaying alone; but, besides, it applies directly and with equal advantage to the study of an ore deposit, to the working of a refractory ore, or to almost any technical problem. When once the spirit is gotten, such study is most fascinating.

One of the most valuable aids in this method of study of any art is the keeping of notes which fully, clearly and accurately describes the *important points* of what has been seen and done. These notes ought by all means to be written at the time and on the spot, which gives accuracy and facility in writing the description. Therefore take your note-book into the furnace room and write your notes where and while you are working. Describe what you yourself see: you should see enough every day you work to more than fill a page. This does not mean to repeat what is already stated in these sheets. Have a hard pencil in your pocket, and write the notes in once for all. Write the notes legibly and keep the book as clean as you can conveniently; then do not mind a little coal dust or an occasional acid spot. The notes of laboratory work for each day should be completed in full before leaving for the day whether or not numerical results have been obtained. You should stop work soon enough to have time for doing this and for cleaning up. So much for notes taken in the laboratory.

Now as to library notes. The best literature of assaying is widely scattered in many text-books, as well as in periodicals and in transactions of engineering societies. We use the two books which seem the best, on the whole, as text-books; but you should by no means confine your reading on assaying to these. Numerous references will be given as we go on and you will find it of value to make abstracts in your laboratory note-book of what seems to you of most interest and use in them. Many most valuable papers on assaying have appeared in the Trans. A. I. M. E. and these may be obtained of the secretary at small cost and bound into the note-book. By the free use of these methods in both library and laboratory you can construct for yourself what will be, perhaps, the best text-book possible.

For these papers, address Dr. R. W. Raymond, Lock Box 223, New York City.

After finishing each assignment, report that fact, presenting the note-book record of the work done for correction and criticism.

CLASSIFICATION OF ERRORS.

No measurement can be made with absolute exactness, whether it be the measurement of a physical quantity in the physical laboratory or by a surveying instrument in the field or a measurement of a chemical quantity by the wet methods of analysis; although many measurements can be made, as you may have found already by previous work in surveying, physics or chemistry, with extreme precision, so that the error becomes vanishingly small, and so that any degree of approximation desired can be attained by a corresponding expenditure of money, time or skill, one or all. This criticism applies with equal truth to the methods of fire assaying.

In assaying, as in all other measurements made for technical purposes, where time and expense must be considered as well as accuracy, it is of great importance to locate whatever errors there may be, to determine their amount and to study methods of minimizing them. Some errors can be entirely eliminated, while other errors cannot be eliminated, although in any reliable method they can be rendered both small and constant by care and skill.

The errors which may enter the result in fire assays may be grouped under four heads:

1. Errors caused by interchanging the samples or by interchanging the assays in the furnace, or during parting and weighing, thus confusing results, in mistaking the weights used in weighing the ore, the silver or the gold, or numerical errors in calculation. The weights used in assaying have been so simplified that errors of calculation can be made only by gross carelessness. Such *blunders* are as inexcusable in a beginner as in the most experienced; they generally lead to much greater and more serious errors than are likely to be made in any other way, and often to ludicrously wild reports. It is very important for your future success that you should use great care not to make any of these blunders.

2. Errors of manipulation caused by lack of care or of skill. Errors of manipulation occur most commonly as mechanical losses, but they may enter also from the use of too high or too low a heat in the furnace, from the application of a certain heat too early or too late, from the use of fluxes or reagents in improper proportions or from other causes. Errors of this class can be banished, of course, by the use of care and of skill; it will be the purpose of the work of the course to give you the special knowledge and the training of the eye to see and of the

hand to do which constitute this skill. Written and verbal instruction can and should explain the principles, and describe the methods which have been found in the practice of the instructor himself and of others to yield the best results; but for the mastery of the small but valuable details of assaying, as of all arts, the careful, intelligent, critical, progressive practice of that art by the learner is necessary. Therefore be constantly on the alert to discover the points of defective manipulation in the work as you perform it which are likely to introduce errors and to adopt such improved methods as shall give your work the greatest accuracy, certainty and speed.

3. Errors caused by imperfection of apparatus or materials, which we may, for the lack of a better term, call "instrumental errors." The best bead balances can be relied on to weigh only to .01 mg., or at most ½00 mg., and may be less accurate than this. The milligram weights sold are often, or even generally, not standard or even consistent among themselves to .01 mg. The lead and litharge used always contains a little silver, although such care should have been taken to purify assayer's litharge and test-lead that it contains only a little silver. I have found from .05 to .90 mg. of silver in 100 grams of these reagents as purchased. With the apparatus sometimes chosen for parting, and the method used, a loss of gold can take place unnoticed. If the nitric acid contains chlorides, silver chloride will be precipitated on the gold and weighed as such. Errors of this class can be entirely eliminated or made so small that they can be neglected by a proper choice of apparatus and reagents.

4. Errors which proceed from defects in the method itself as an analytical process, or "sources of error", properly so called; e. g., a "source of error" in determining lead gravimetrically as sulphate is due to the slight solubility of lead sulphate. We shall discuss errors of this class occurring in fire assays fully later in the course: it is not profitable to do so now. Here it need only be said that the sources of error for gold are very small, and for both gold and silver quite constant in skillful hands.

SYSTEM, ORDER AND CLEANLINESS.

To avoid errors and to accomplish the most work with the least effort, and in the shortest time, system, order and cleanliness are indispensable. You should train yourself to good habits in these matters at the outset.

SYSTEM.

An assayer is generally required to carry on at the same time from 4 to 50 assays; and as each one has to pass through about a half dozen operations, a simple system must be adhered to in keeping track of each to avoid confusion. Here we shall adopt the following system, which is the same as or like that generally used in practice by assayers at smelting works and by others.

1. The *ore samples* in this laboratory are given running numbers, never to be repeated, so that no two samples bear the same number and samples can be designated by number.

2. Number your *assays* each day from one upward, letting duplicate assays bear different numbers; thus your assay numbers February 1st will run from 1 to 4, and February 3rd from 1 to 10, if you make 4 assays February 1st and 10 Feb. 3d.

3. Enter in your tabular record of weights and results, before commencing the assay, the date, the number and mark on the sample and your day assay number for each assay. Enter immediately in this table in a neat and permanent form the weights and results as they are obtained.

4, To avoid confusion, keep the assays on the trays, in the parting cups and in the furnace in 1, 2, 3 order of the assay numbers. In addition, colored chalk which will not burn off is provided, with which to place on scorifiers, crucibles, cupels and trays the day assay numbers, if you think necessary.

ORDER.

A place is provided for every tool; all tools are in the places where they can be most conveniently used; hence form the habit of *taking your work to the tools*, not the tools to your work.

Please see to it before you leave the laboratory for the day that:

1. Your fire is drawn;

2. That your private tools are put away in drawer, locker or closet; that any apparatus you have used is clean, in good order and in its proper place.

Both your own work and that of the others working in the laboratory with you will be hindered if tools are left out of order; and disorderly surroundings easily produce confusion of mind.

CLEANLINESS.

Remember that fire assaying is only a special branch of quantitative chemical analysis.

Attain in your work, first of all, accuracy of manipulation and a clear understanding of the methods and the reasons for what you are doing; but also work with the idea of accomplishing everything in the shortest, quickest way. After we have been over the methods for a group of different ores, miscellaneous samples will be assigned to give you practice in carrying through a number of assays on a variety of ores at the same time.

ASSIGNMENT I. PRACTICE IN ACCURATE WEIGHING

Pure gold is worth $602,928.47, or about $603,000 per ton.

A substance containing	1%	of gold is worth	$6,030	"	"
" "	.00001%	" "	.06	"	"
" "	.00004%	" "	.25	"	"

It is impractical, in general, to take more than 1 assay ton, equal to 29,166 milligrams, of ore for assay in one charge, and with some ores and some methods only 1-10 of this weight can be taken.

We must not be limited by our bead balance to an accuracy of less than $.25 per ton or .00004% in gold, using one assay ton of ore.

.00004% of 29,166 milligrams equals .012 milligrams. We must, therefore, be able to weigh the gold to .01 milligram. This is the limit of weighing practically attainable, with good assay balances in proper adjustment in every day work; for some $200 balances an accuracy of 1/100 mg. is claimed.

To weigh to .01 milligram with certainty requires a good assay balance and skillful manipulation. Since the certain and precise weighing of the parted gold quickly is a matter of absolute necessity for close work, we shall in what follows take up at some length the theory of the balance and the practical details of its construction, adjustment and use.

THE THEORY OF THE BALANCE.

EXPERIMENT—The balance being in adjustment, so that the pointer rests at the centre of the scale when the pans are empty, place a small piece of gold, not large enough to cause the pointer to be deflected off the index, in the left pan and allow the beam to vibrate. It will be found that the amplitude of the vibrations gradually decreases and the vibrations finally cease, the resistance of the air and friction on the edges bringing the balance to rest. Note the new position of the pointer.

What is it which now holds the weight of the gold in equilibrium?

It is this: The centre edge of a balance is always placed very slightly above the centre of gravity of the beam. Now when a small weight is placed in the left pan and the beam is allowed to vibrate until it comes to rest in the new position, the centre of gravity (refer to Figure 1) will move to the right from c to c′ and the weight of the beam, now no longer directly under the axis of support, will tend to turn the beam in the opposite direction from that in which the weight of the little piece of gold in the pan tends to turn it.

Notice that the more the beam is deflected, the less leverage will the weight of the little piece of gold have in causing the beam to turn in one direction and the more leverage will the weight of the beam have in causing it to turn in the opposite direction. Hence you can perceive that whatever the weight of the gold(provided it is less than, say, one-fourth milligram) the weight of the beam will hold it in equilibrium in the same way but that the beam will be deflected less for a light than for a heavier piece.

From the facts stated and from the figure, you can now see that the deflection will be *greater*, the greater 1. the excess of weight in one pan over that in the other. 2. The length of the beam; and that the deflection will be *less*, the greater 1. the weight of the beam, 2. the distance between the centre of gravity of the beam and the centre knife edge, or its axis of support.

These facts may be accurately and concisely expressed in mathematical language as follows:

Let a = the angle through which the beam and pointer are deflected.

Let p = the excess of weight in the left pan over that in the right.

Let l = the distance between each outer edge and the centre edge, or one half the length of the beam.

Let G = the weight of the beam.

Let d = the distance between the centre of gravity of the beam and its axis of support.

Then for the position into which the beam has been deflected by the weight p, we may write the equation of moments:

pl cos a = Gd sin A.

(Dividing both members of this equation by Gd cos a, we have)

$$\frac{pl}{Gd} = \frac{\sin a}{\cos a} = \tan a$$

Since a is always a small angle, we may write

$$a = \frac{pl}{Gd}$$

or a is directly proportional to p and l and inversely proportional to G and d.

The Sensibility of a balance may be defined as the number of divisions on the ivory index through which one milligram will cause the pointer to be deflected; or, expressed in another way, the Sensibility is the weight in hundredths of a milligram which will cause the pointer to be deflected one division on the index.

Hence the sensibility is greater the greater the length of the arms and the less the weight of the beam and the distance of its centre of gravity from the centre edge.

EXPERIMENTS—

Draw the diagonals accurately on a card; their intersection will pass through its centre of gravity.

1. Support it by piercing it with a pin vertically above its centre of gravity when the long side is level. Set it in vibration. It is now in stable equilibrium.

2. Pierce it below its centre of gravity. It is now in unstable equilibrium.

3. Pass the pin through its centre of gravity and attempt to vibrate it. It is now in neutral equilibrium.

4. Move the bob on the pointer of the balance upwards, then vibrate the beam; note that this increases its sensibility and causes it to move slower.

5. Move the bob down and note the opposite effects.

6. Move the bob as high as possible. This will throw the beam into unstable equilibrium. Attempt to make a weighing.

7. Set the bob at such a position that one tenth milligram moves the resting point exactly 2½ divisions on the index.

CONSTRUCTION AND ADJUSTMENT OF THE BALANCE.

The following details of construction and adjustment should be fulfilled in a balance intended for gold assaying:

1. The "knife edges" should be true and so sharp that a strong (say a Coddington) lens will not show any flatness on the edge where it touches the plane. Many knives are ground hollow in the centre as seen on a side view, so that the plane touches at only two points. Should a lens show the bearing

edges or points of an old balance to have been worn dull or to have rusted badly or to have been injured, they should be re-sharpened; only a skillful balance maker is competent to do this.

2. The beam should be light in order to give the balance greater sensibility. This can be done, since an assay balance should be loaded with not more than two grammes in each pan. The beam of an assay balance should not weigh more than 20 grams, and preferably less than half of this.

3. The centre of gravity of the beam should be near its point of support. Most balances have a bob sliding on the pointer, by means of which the sensibility can be adjusted; when the 1st, 2nd and 4th conditions are fulfilled, a deviation of one division on the ivory scale should correspond to .04 or .05 milligram; but if these conditions are not fulfilled, the balance is apt not to give consistent weighings with this high degree of sensibility. Unless the beam is light, it is apt to be somewhat "top heavy," and the time of vibration will be inconveniently long, both due to the centre of gravity of the beam being brought so near to the axis of support. On the other hand, it is convenient, with a light beam, to have the balance quite sensitive, since then a slight difference in weight produces a large deviation along the ivory scale, which can be easily read.

4. The balance should be mounted on an absolutely stable foundation, so that no vibration can be communicated to it. Such a support is most simply and cheaply obtained by placing it on top of a post, one end of which is buried in the ground and which comes up through the floor without touching any part of the building; or by placing it on a shelf fastened to a brick wall by substantial brackets and independent of any other part of the building. If the balance has four screw legs, they must all touch the table so that it cannot rock on two of them. The door must slide freely without sticking.

5. Adjustment. The pan and beam supports of the balance should be so adjusted as to throw the balance into action when the key is turned without any jar or shock which would tend to change its resting point. The beam supports should be so adjusted that the pointer will stand at the centre of the scale when the balance is out of action, and also so that the beam can rock very slightly on the supports. The pan supports should barely touch the pan hanger when the balance is out of action, so as to support it firmly when putting beads and weights on

and off; but should not be so high as to cause any buckling or movement in the pan hangers on turning the key. The beam should not stick in the slightest on either side when released. The balance should, of course, be set up level and put together according to prick marks.

6. The balance should be placed in a light place where the direct rays of the sun cannot shine on it and it should be covered with a case or box when not in use, as a dust protection. Every two or three months the beam should be dismounted, the knives and planes brushed with a camel's hair pencil, and the dust wiped out of the inside of the case.

THE USE OF THE BALANCE.

1. Turn the key slowly, especially in lowering the beam or throwing the balance into action. Bear in mind that each time this is done, the "knife-edge," which should be so sharp that a strong lens will not show any flatness on it, supporting a beam of quite considerable weight, impinges on a hard agate plane with an appreciable velocity even when the key is turned slowly; and that this slight blow is repeated thousands of times in the course of years. A balance with knife-edges should not, however, in general need to have the knives re-sharpened oftener than once in ten years, with careful use.

2. Have the amplitude of swing not more than three or four divisions.

3. Avoid air currents in the case when reading.

4. Be careful not to give the instrument any shock which might alter its resting point while using it.

5. The position on the index at which the pointer would come to rest, which is called the resting point, can be noted more accurately as well as more quickly from readings taken while the beam is swinging than by reading after it comes to rest. Since the amplitude of the swing is constantly decreasing, the resting point is not the average of a reading on the left and the next on the right; but is the average of the reading on the right with the mean of the reading when the pointer started at the left and when it returned; e. g., if the first reading on the left is 2.5 from the centre, on the right 2.9 and on the left 2.3, the resting point is ¼ of a division to the right of the centre. If the number of hundredths of a milligram corresponding to a deflection of one division on the index is known, the rider can be set at the second trial so that it will balance

the weight of the gold and the new resting point will be at the centre.

Read at this time:

1. Aaron's Assaying, Part I, pages 24-34 and 82-87.
2. Furman's Assaying, pages 51-54.
3. Dealers' catalogues descripive of the balances made by Oertling, Becker, Troemner, Ainsworth, Kohlbusch, and Smith & Thompson. Study the details of the mechanism intended to make each balance sensitive, rapid and consistent in giving the weighing, and convenient in use. Note the prices.

Which should you consider the best balance for a small gold mine where the price must not exceed $100? which for a smelter or a large laboratory where the best balance is desired regardless of the price? State your choice, with full reasons in each case, in your note-book.

It would be well to secure the catalogues of John Taylor & Co. and the Justinian Caire Co. of San Francisco and of the Denver Fire Clay Co. and Smith & Thompson of Denver and others for your library for future reference.

THE PRINCIPLE AND USE OF THE RIDER.

Weights of less than one milligram, or, at the least, one-half milligram, are too small to be easily handled; hundredths of a milligram are therefore weighed by the use of a "rider." This is a bit of fine aluminum wire which can be placed at any point along the beam, the latter being divided into equal divisions from the centre to the end knife edges (look at the balance), so that the fractional weight can be read off by precisely the same principle as from the pea of a steelyard.

If the rider be placed over the centre edge, it will, of course, have no effect in turning the beam; if placed over one of the outer edges it will have the same effect in turning the beam as if it were placed in the pan below and will, therefore, hold in equilibrium a weight equal to its own in the opposite pan. If placed halfway between the centre and one of the outer edges, it will balance one-half its own weight in the opposite pan; and in general, it will, acting on the principle of the lever, if placed anywhere on the beam, balance the same proportionate part of its own weight in the opposite pan as its distance from the centre edge is of the whole distance between the centre edge and one of the outer edges. Hence to read the weight from the rider, we must take into account both the weight of the rider and the number of graduations on the arms of the beam.

1. Weigh your rider against a milligram weight in one of the pans.

2. Set the rider at 0; 1.00; 0.50; 1-10; 7-10; 0.72; 0.39; 0.05.

Riders of assay balances should not exceed two milligrams in weight, so that the graduations for each 1-50 or 1-100 milligram will be of considerable size. The rider must be of standard weight.

The most convenient modern balances (called "open beam" balances) have the beam of such a shape that the rider can be set over the centre edge, over either of the outer edges or at any intermediate point; but in some balances the rider can be set on only certain portions of the beam.

It is sometimes convenient to use a second rider, besides the one with which weighings are read, at a short distance to the left of the centre of the beam. By shifting this slightly, the resting point of the balance when empty can easily be kept at the centre of the index.

1 Weigh four small pieces of parted gold separately, putting each piece into a clean capsule after weighing. Then weigh all the pieces together: this weight must check the sum of the separate weighings to 0.01 or, at most, 0.02 milligram; if it does not at the first trial, repeat until you are able to make it check.

2. Test your milligram weights up to 10 milligrams to determine how much in error each is from being consistent and standard. Also test the 1000 and 500 milligram pieces against the smaller weights.

WEIGHING BY DEFLECTIONS.

Since from the theory of the balance the deflection of the beam is proportional to the excess of weight in one pan over that in the other, it follows that, if we know how many divisions deflection a piece of gold produces and how many divisions deflection one milligram produces, we can determine the weight of the gold without the use of a rider or weights.

By the use of this method it is possible to obtain fairly accurate results, even with a balance which is not very sensitive. Some cheap balances without riders, and especially balances which have been damaged by misuse, are to be found at small mines where not much work is done; and it is well, therefore, that you should learn to obtain the best results possible under these conditions, as you may be called upon to use such a balance. But a good balance with a light rider should be obtained

as soon as possible. Since it is impossible to determine the resting point nearer than 1-10 of a division of the ivory scale in reading it from a single swing of the pointer, as is done in the method of weighing with a rider, it follows that any assay balance with which the gold is weighed and a rider used should have a sensibility equal to 0.10 milligram for one division of the ivory scale or a sensibility greater than this. For the sake of convenience and rapidity of weighing, the sensibility of the finest balances should be .04 or .05 of a milligram for one division of the ivory scale. Moreover, practice in this method will give skill in the use of the balance. Balances are sometimes improperly used, through a lack of knowledge of the theory of the balance, with such a heavy rider or with such a low sensibility that it is impossible to weigh the gold with the precision required in working, especially, on small amounts of ore or with low grade material.

In reading, call the graduation on the extreme left of the index 0, at the centre 10, and at the extreme right 20. The following example will show the method of making the readings and calculations:

Pans Empty:		Gold and weights on pan:	
7.8	11.9	8.7	13.9
7.9	11.8	8.9	13.7
8.0	11.7	9.1	13.5
8.1		9.3	
	3 | 35.4		13.7
4 | 31.8	11.80	4 | 36.0	9.0
7.95	7.95	9.00	
			2 | 22.70
	2 | 19.75	Resting point loaded	11.35
Resting point	9.88		
		Resting point empty	9.88
		Deflection	1.47

Suppose the balance has been found to have a sensibility of 0.21 milligram for one division. 1.47×0.21=0.31 milligrams.

1. Set the bob on the pointer at such a position that the balance has a sensibility of about 0.20 mg. for 1 division, or so that 1 mg. produces a deflection of about 5 divisions.

2. Determine the resting point of your balance by several trials until separate results do not vary more than 0.05 division of the ivory scale. Take an even number of readings on one side and an odd number on the other, and take the least num-

ber in each set which will make the separate results agree well.

3. Determine the deflection for 1 mg.

4. Fill out the following deflection table, carrying the weights to .001 milligram:

DEFLECTION TABLE

Div.	Mg.	Div.	Mg.	Div.	Mg.
0.1		0.4		0.7	
0.2		0.5		0.8	
0.3		0.6		0.9	
				1.0	

5. Weigh four pieces of parted gold, first separately and then together by this method.

TABLE OF TROY WEIGHT

Pounds	Ounces	Pennyweights	Grains
1	12	240	5760
	1	20	480
		1	24

Gold and silver bullion is always weighed in Troy weight in England and in the United States. In England it is weighed in ounces, pennyweights and grains; in the United States in ounces and hundredths. In France and Germany it is weighed in grams.

In the United States gold, silver, lead and copper ores are weighed in short tons of 2000 lbs.

One pound Avoirdupois contains 7000 Troy grains.

One gram contains 15.43 (plus) Troy grains.

According to the U. S. coinage laws:

One Troy ounce of pure gold is worth $20.67 (plus)

One Troy ounce of pure silver is worth $1.2929, measured in U. S. coin.

1000 parts of U. S. gold coin contain 900 parts pure gold, 100 parts copper by weight.

1000 parts of U. S. silver coin contain 900 parts pure silver, 100 parts copper by weight.

From the above data, solve the following problems in your note-book:

1. What is the value of one gram of pure gold?

2. Calculate the weight in both Troy grains and fractions of an ounce of a $20.00 gold piece.

3. What is the value in gold of the silver in a U. S. half dollar when the market value of silver is 61 cents an ounce?

a. Rolling out Bullion.

Roll out a dime so that it can be conveniently cut up with shears. Anneal during the rolling whenever it becomes hard and elastic, by heating to dull redness, and then quenching in water or allowing it to cool in the air. Anneal again when the rolling is finished, clean with sand paper and preserve for practice in cupellation. The dime should be rolled out somewhat thinner than an ordinary card.

b. Making Cupels.

Make 100 good cupels and keep them for your own use.

A good cupel must be perfectly smooth on its interior and of the correct porosity so that it will have the right absorbent power for litharge. It must not crack when heated in the muffle and should be so firm that it is in no danger of crumbling in the fingers or in the tongs. Good cupels give out a more or less metallic ring when two of them are slid into shape impact after being air-dried. They should, when air-dried, be of such firmness that they will not break when dropped from a height of two feet onto a hard surface.

The most necessary requirement for making good cupels and making them rapidly is to have the "dough" of just the right dampness and well kneaded. It must be so moist that it will adhere like dry snow when squeezed in the hand and must have lost entirely the dry, prickly feeling which it has before mixing; it must not, on the other hand, be so wet as to be at all soggy. When damp enough, it will not be perfectly white but of a grayish tinge. It is well to mix the dough a little while before you begin to mould the cupels. It may be told at once whether the dough is of the right dampness by making one or two cupels: if too dry, the cupels will be somewhat crumbly and will be hard to get out of the mould; if too wet, the interior of the cup of the cupel will be more or less rough, caused by a little of the boneash adhering to the pestle. Somewhat different effects in these respects will be obtained according to the force of the blows struck and the resulting degree of compression. It is better to err on the side of making the dough a little too dry than too wet. The bottom of the pestle should be perfectly dry and clean after the cupel is shoved out of the mould.

The boneash put into the ring should be pressed down firmly and evenly all about the ring with the fingers before

driving in the pestle; otherwise the cupel will not be evenly compressed in all parts of it by the pestle.

A dilute solution of potassium carbonate is sometimes used in making cupels but water is about as good. If potassium carbonate solution be used, it must not be too strong or the cupels will not absorb litharge well.

Cupels ought to be air-dried for several days, or better a fortnight, before use, preferably in the sun for a few hours of this time; but if properly made, they can be used immediately in an emergency. As they still contain some moisture after being air-dried, no matter for how long, it is necessary to let them remain at a bright red heat in the mffle for at least ten minutes before adding the buttons to be cupelled; otherwise the steam forced through the molten lead bath will cause the cupel to "spit", occasioning mechanical loss.

Even good cupels are apt to crack if heated above a low yellow, or if taken from the furnace, allowed to cool and again heated.

Cupels should be heated up as slowly as convenient to lessen the chance of their cracking. It is well to put as many as will be needed during the day into the back of the muffle when the fire is lighted, if you intend only to cupel; otherwise they may be heated for 5 or 10 minutes in the front of a hot muffle, then pushed back. Any roughness around the rim of the cup should be rubbed off before putting the cupel into the muffle to prevent bits of boneash from falling into it.

c. Furnace Repairs. Putting in Muffles.

Lime mortar and hydraulic cement do not answer for use with masonry exposed to flame and high heats, which cause them to fuse and crumble. The lime is replaced by fire clay, sometimes mixed with sand and sometimes with powdered fire brick. Fire clay is a hydrous silicate of alumina, as free as possible from iron and the alkaline oxides and forms a plastic and very sticky mortar when wet. On heating, the fire clay loses its water and its plasticity and the mortar hardens.

A mortar of fine texture, such as that made of lime and sand for ordinary brick-work, hydraulic cement for use under water or fire clay mixed with fire brick dust for furnaces is needed for uniting well-fitting surfaces such as those of brick; but where larger masses of the mortar are used, as in concrete, broken rock is mixed in with the cement, or in luting around the mouth of a muffle, broken fire brick with the fire clay mor-

tar, to make the texture coarse, as this adds strength and prevents cracking on drying.

Any mortar should be thoroughly mixed, sticky and of the right consistency for the particular work. It is well to mix the mortar several hours or days before use. The bricks and the brick-work should be thoroughly wet before applying the mortar, for their surfaces absorb water with avidity and if the mortar dries too quickly it will be less strong. Even though the mortar be of the best, use as little of it as possible, since the brick, which have been kiln-fired, are much the stronger. The mortar should be rammed firmly between the bricks, so as to fill every crevice. The best way to attain these two things is to put an extra amount of mortar on the wet brick and ram or drive it firmly into place, letting the excess squeeze out. The fine mortar used in laying up bricks should be wetter or thinner than the coarse mortar used in large masses.

Assay and other furnaces are generally built of red brick laid in lime mortar, lined on the inside with one course of fire brick laid in fire clay mortar. Red brick would fuse and lime mortar crumble at a high heat. No iron surface will endure a strong flame with excess of air for more than a few hours unless cooled by a water-jacket, as in a steam boiler or a water-jacketed smelting furnace. The grate bars are protected because they are under the fire and also are cooled by the cold air passing between them on its way into the fire.

Brick are cut to special shapes by the use of a sort of hammer, called a scutch, with a chisel edge, the brick being held in the hand; or by the use of a broad, blunt chisel, the brick being placed on an even spot of moist ground to get a good support.

The ash left behind by the burning of the coke is rich in alkaline and other carbonates, which cause the ash in part to frit with the walls of the furnace and form clinkers on them. These can be cut off quite easily with a heavy iron or steel bar, chisel-shaped at one end, while the furnace is still hot, after drawing the fire; on cooling, they become very strong and adhere tenaciously to the brick, so that there is great danger of breaking off pieces of the walls of the furnace by removing them then. They should be broken off, with care not to injure the furnace walls, just after a hot fire has been drawn, when they accumulate to such an extent as to obstruct the working down of the coke around the muffle.

In putting in a new muffle, first clear out the old muffle (breaking it if necessary) and its luting and any bad clinkers,

with care not to injure the brick of the furnace; and put the grate bars in place. Bore two holes in the back of the new muffle, each ⅜ of an inch in diameter, 1½ inch above the bottom of the muffle, using the tange of a file. Make some coarse fire clay mortar by crushing pieces of brick to pass a 4-mesh screen, and mixing this with fire clay and water to make a mortar which contains as much of the coarsely broken brick as it can and still be very sticky, not gravelly. The muffle should be placed as far in as possible and still leave it a good bearing on the front wall of the furnace; its bottom should be level crosswise and slope upward slightly toward the back. It is well to place a small piece of brick between the rear wall of the furnace and the muffle to prevent it from slipping farther back. New furnaces have a brick projecting from the rear wall to support the back of the muffle; if this is lacking, cut a brick of the proper length, put mortar on one of its narrow sides and drive this side firmly against the rear wall, the bottom of the brick resting on the grate bars. Wet the brick-work where mortar is to come in contact with it. Stop the space between the mouth of the muffle and the furnace by pressing into it very firmly as much of the coarse mortar as you can, pressing it outward from the inside of the furnace. This mortar should not be too thin, and it should be compressed between one hand on the inside and the other on the outside of the furnace. Leave the doors of the furnace open for the mortar to dry, clean up the furnace and the tools used and return all tools to their places. The furnace can be used immediately in case of necessity, but it is preferable to allow the mortar to dry for a day or over night. A new muffle should be heated up slowly the first time.

ASSIGNMENT 3. PRACTICE IN CUPELLATION.

a. Cut off a piece of Peruvian silver coin weighing 45-55 milligram, weigh it to 0.1 milligram, (do not waste time in weighing it to .01 milligram, since your errors at the furnace will be considerably greater than 0.1 milligram,) mg.,) and wrap it in about 15 grams of sheet lead, weighed roughly on the pulp scale. Prepare as many of such assays (up to 10) as you have time for. Record the weight of each properly in your tabular form and place your assays in the parting cups in such an order that you can identify them. Take the box of parting cups to the furnace and cupel in sets of two each, in order that you can repeat the operation a number of times for practice. Record in your notes, for each assay, any variations from the proper heat.

Beads are to be cleaned and put in the cups in the bead tray; the cupels are then to be thrown away and the cupel tray returned to its place; the box of cups containing the cleaned beads is then to be taken to the balance room for weighing.

Open the draughts, light the kindlings and place in the back of the muffle a half dozen cupels (or as many as you will need) and close the muffle door. Put on about one-third of a hod of coal; when it is well lighted poke it down and fill the furnace with coke up to the bottom of the flue. Poke the coke down solidly under the muffle as soon as it is bright on top and add more if necessary, so that the muffle is well covered. Allow the heat to rise a little above a bright red, then let the muffle cool until it is at the right heat (a bright red about five inches from the front) to put in the leads. Some red should show in the back of the muffle.

Cupel in sets of two each, as nearly as you can at the proper heat, as demonstrated to you previously, "feathering" the cupels without "freezing" the assays, if you are able. Refer to Brown's "Manual of Assaying," pp. 214-223, for a description of the proper heat. Finish the assays at a somewhat higher heat, increasing the temperature by shoving the cupel back in the muffle, by increasing the heat of the furnace or by laying a hot piece of coke before the cupel a minute or two before the play of color comes (depending upon the heat of the cupel) so that the heat is a bright red *when* the play of color comes. The colors should move rapidly over the bead and be succeeded soon by the pure silver color, when the cupellation is finished. Examine the beads to see that they are well rounded and slightly corrugated or "frosted" on the bottom but free from rootlets. If they are smooth or are pitted on the bottoms, or if

they show rootlets, they contain a little base metal. Weigh the beads and record results in tabular form as follows:

FORM OF RECORD.

Date	Assay No.	Coin	Lead	Wt. Bead	Silver Fineness	Remarks
Feb. 1	1	49.5	15	43.6	088.0	

Nov. 1, 1899.

Assay No. 1. Cupelled at proper heat for 10 min., then "froze"; remelted. Brightened at proper heat, etc.

No. 5 and No. 6. Driven at the proper heat so that considerable feather litharge formed; beads well rounded and bottoms good.

Dissolve 25-50 mg. coin silver in a little nitric acid and test for copper with ammonia. Do the same with a well brightened bead. Record results.

The chemical reactions which go on in the cupel are:

Pb plus O = PbO (litharge, fluid at a bright red heat.)

PbO plus Cu_2 = Cu_2O (infusible) plus Pb.

The Cu_2O is dissolved by the fluid litharge and the two are absorbed by the porous cupel, as the soil absorbs water.

This reaction also takes place to a *very small* extent:

Ag_2 plus PbO = Ag_2O (which is also carried into the cupel.)

This is the cause of a slight loss of silver by "cupel absorption."

REFERENCES ON CUPELLATION.

1. Furman's "Assaying," page 128.
2. Brown's "Manual of Assaying," pp. 214-223.
3. Bodemann & Kerl's "Assaying," pp. 119-124.
4. Mitchell's "Mannal of Assaying," pp. 617-631, 6th ed.
5. Silver Losses in Cupellation. Godshall. Trans. xxvi-473

b. A repetition of assignment a, except as follows: Prepare 4 assays, each to be composed of 100 to 300 mgs. of coin and 15 gms. of lead, and cupel in sets of two. These beads will be of such size that there will be danger of their "spitting" unless removed cautiously from the muffle so that they shall cool gradually. It is well to cover the cupel with another hot one and allow both to cool near the mouth of the muffle. All danger of "spitting" has passed when the inside of the cupel has reached an incipient red. Fix the heat colors indicating a good cupellation temperature in your mind so that you will not forget them, as we now pass on to the making of scorifications

at a higher heat, this being the last practice exercise on cupellation of ore assays without using coolers.

c. Cupellation in a large muffle, with the use of coolers.

You should have a substantial fire before beginning a cupellation; the furnace should *appear* hotter than in the small coke furnaces in order that the buttons may melt quickly. As soon as they have melted well, the temperature must be lowered at once so far by putting cold scorifiers on top of the cupels that the cupels shall appear quite a dark brown inside and feather litharge shall be seen forming in the cupels. Good results will be obtained by placing 2½ inch scorifiers on the cupels in the back of the muffle, 2¼ inch over those in the centre and 2 inch over those in the front. The coolers must be changed if the heat runs up after they begin to get hot and hence to lose their effect. It is well to put low pieces of glowing coke in the mouth of the muffle to keep the temperature more uniform from front to back. To brighten the beads, take out the coolers, and open the draughts if necessary. During the driving, the draught in the muffle should not be too strong, but the fumes should be drawn partly to the back and should in part escape by the front.

Use old assay buttons of about 15 gms. or add a bead to each 15 grams of scrap lead. Do at least 16 cupellations at a time. Do as many sets of 16 as necessary to give you a mastery of the furnace and the method. After drawing the fire, close all the lower doors and open the charging door. By the use of coolers you should be able to feather all the cupels and have all the beads well brightened. Coolers do not control the heat as satisfactorily in small muffles.

d. Assay of Base or Silver–Lead Bullion.

Read before beginning the work:

1. Furman's "Assaying," pp. 14-18 and pp. 232-236.
2. Brown's "Assaying." Appendix, pp. 418–421.

"In base or lead–bullion cupellations, the feathers should extend all the way down the inside of the cupel, almost to the silver button in the bottom. The silver, however, must be clean, round and bright. The secret of cupellation (using a soft-coal or flame-heated muffle) lies in having a good, hot bed of fire and in keeping the muffle cool, *very* cool, by coolers. Then at the end, take out the coolers, open the draughts, and if the fire is *alive*, the heat will come up at once and your buttons will all blick as nicely as could be desired."

Save the beads after weighing to be parted for gold later.

ASSIGNMENT 4. FIRE ASSAY OF SILVER BULLION.

Read before beginning the work:

1. Furman's "Assaying," p. 237.
2. Aaron's "Assaying," Part II, pp. 42–45.

We have hitherto used an excess of lead in cupelling silver coin to imitate the conditions in an ore assay and to give you more practice in running the furnace. In an assay of bullion, however, it is necessary to use only the proportion of lead required to free the cupelled bead from copper, as found by experiment for bullion of each fineness and given in tables which are to be found in the above books; e. g., 7 parts of lead to 1 part of bullion which is 0.900 fine in silver. See Furman p. 238. It is customary, also, to determine the loss of silver during cupellation by means of a check assay, whose composition is known, called the "proof centre," and to make a correction accordingly, as explained in the books above referred to.

The fusibility of assays with ½ gram of bullion and 3½ grams of lead is so much less and the beads so much more difficult to brighten properly that it will be well to do two or three for practice, one at a time, before making the assay proper. The accurate execution of the fire assay of silver bullion requires a nice adjustment of the heats and a considerable degree of skill. To get this practice, weigh three or four assays of the Peruvian coin, each exactly 500 mg. (within 0.1 mg.), and 3500 mg. lead, and cupel them as carefully as you can in the muffle. If properly done, the bead will be of a bright silver white, will not have flat edges, and the result without corrections will be 0.896.

For perfect work, where as much as three or four grams of lead are used, a slight ring of feather litharge should form in the driving, which should almost or quite disappear in the brightening; but you must work carefully to get feather litharge without freezing the assay, using this small amount of lead. The assays must be brightened quite hot so that the beads will be silver white and not have flat edges. A somewhat higher heat than you have been accustomed to is necessary all through, on account of the large proportion of silver which the assays contain.

Sample 57. Samples A and B are duplicate samples of a bar of refined silver bullion.

Make a preliminary assay with about 500 mgms. of bullion to learn the approximate fineness, that you may know how to make up your proof centre and how much lead to use. Correct

the results of the preliminary by adding three to seven points, according to what experience has taught you to be the cupellation loss under these conditions; then carry out the assay proper as described, using the heats as just learned on the coin bullion. Weigh duplicate assays, each 500 milligrams, and cupel them as carefully as you can on the same line in the muffle as the proof centre, one on each side of it.

Record results in some such form as this:

	Sample A	Proof Centre	Sample B
Bullion	500	——	500
Silver	——	xxx.x	——
Copper	——	xx.x	——
Lead	3,500	3,500	3,500
Cupelled Bead	xxx.x	xxx.x	xxx.x
Cupellation loss (thousandths)		0.0xx	
Silver fineness	0.xxx		0.xxx

Additional references:

1. Mitchell's "Assaying," pp. 631–634.

2. Bodemann & Kerl's "Assaying", on the "Fine Silver Assay," pp. 139-150.

ASSIGNMENT 5. PARTING OF GOLD AND SILVER.

Gold is insoluble in nitric acid; silver is acted upon readily, silver nitrate going into solution and oxides of nitrogen being evolved. If we substitute for the pure silver any gold-silver, or "dore" alloy, it is found that all except a trace of the silver will still always pass into solution on proper treatment with the acid and all of the gold remain, provided that the alloy contains not much less than 2½ parts of silver to 1 part of gold. The samples of dore bullion furnished for this assignment all contain at least 2½ parts of silver to 1 part of gold.

After the silver has been dissolved from a dore alloy by the acid, the gold remains as a porous mass which is the more compact and adherent the larger the proportion of gold the alloy contained, the thicker the alloy and the less mechanical disturbance of the gold there is during the dissolving by violent action of bubbles of oxide of nitrogen or of steam. In order to avoid mechanical loss of gold in pouring off the acid and in washing the gold, it should remain in one piece if possible, or at any rate should be as little scattered as may be. The ratio of silver to gold must not, however, be less than 2½ : 1, and with alloys

containing less than 6 to 8 times as much silver as gold, the bead must be flattened to the thickness of a card before parting to insure the acid acting upon the silver in the interior of it. To avoid too violent action of the acid, a dilute acid is first applied to dissolve slowly most of the silver; this is followed by strong acid to ensure the complete removal of the silver.

Chemically pure nitric acid of three strengths is provided at the parting table: 1. Acid of 1.27 sp. gr., made by diluting the acid of 1.40 sp. gr. received from the dealer with ½ its volume of water; this "strong acid" is intended for the second treatment of the gold in all cases, to ensure complete removal of the silver. 2. Acid of 1.16 sp. gr., made by diluting 1.40 acid with 1½ times its volume of water; this "dilute acid" is intended for the first treatment of alloys containing less than about six times as much silver as gold. 3. Acid of 1.05 sp. gr., made by diluting 1.40 acid with seven times its volume of water; this "weak acid" is intended for the first treatment of alloys containing more than about six times as much silver as gold. If the proportions of the alloy are unknown, use the weak acid in preference to the dilute acid for the first treatment.

As gold and silver are found in all proportions in ores, it often happens that there are many times, occasionally over a thousand times, as much silver as gold in an ore. In this case, the gold will separate in a powder or in a very fine filament on dissolving the alloy in acid; but it will be less divided and scattered the more slowly does the dissolving go on, thus weakening the mechanical action of the bubbles of gas, and the thicker and more compact the alloy dissolved, the sphere being, of course, the most compact condition. To keep the gold in one piece on parting beads very poor in gold, use the dilute acid and only warm it slightly, removing from the source of heat as soon as action has commenced; if even 10 or 15 minutes are taken in dissolving such beads, it will be found much easier to pour off quickly without loss. In addition to this, when beads are known to contain only a *very* little gold, in proportion to their silver, they should not be flattened before parting. The gold may be obtained in one piece by these methods even when the alloy contains as much as 4000 times as much silver as gold.

EXPERIMENT.—1. Cut off about 50 mg. of 53, flatten it on an anvil until quite thin, and treat with the 1.16 acid in a parting cup on the hot plate at a good heat so that the silver dissolves quite quickly. 2. Cut off another 50 mg. piece of the same alloy and fuse it to a globule by means of a blowpipe on

an ignited cupel. Treat this with 1.05 acid in a parting cup, warming the acid only gently and removing the cup from the plate as soon as action has begun, so that it takes quite a time to dissolve all the silver; then pour off the weak and add the strong acid. Compare and record results.

DIRECTIONS FOR WORK.

First make sure that your test-tubes and parting capsules are free from chlorides and wash out any specks of dust, that such may not be mistaken for gold after the dissolving has begun. Weigh off two assays of each of the three bullions: for each assay of 51 and 52, cut off a piece weighing within a few tenths of a milligram of 10 mgs., and then weigh the portion cut off accurately to within .01 mg.; for the assay of 53, use about 50 mg. Place one assay from each of the bullions in a parting capsule and fill the cup about one-third full of the dilute acid for 51 and 52 and of the weak acid for 53. Place the three capsules together on the hot plate in the fume closet and heat gently until action ceases. Dissolve the duplicate assays in test-tubes over the alcohol lamp or Bunsen burner in the fume closet, allowing the action to go on slowly in the same way. Now pour off the first acid (containing silver nitrate) into a small, white and clean evaporating dish in a good light, watching sharply to be sure that no minute speck of gold is lost. Add a little of the "strong acid" and heat to the boiling point to ensure complete removal of the silver. Pour off this acid and wash twice with distilled water. Remove the last drop of water from the cups with a glass tube drawn small at one end or with a slip of white blotting paper, dry the cups on the hot plate and anneal the gold by heating to low redness over a Bunsen burner or an alcohol lamp. Having the blotter slightly damp helps to prevent the gold from sticking to it. After washing the gold in the test-tubes with water, fill each test-tube, invert an annealing cup over it, invert the cup and tube so that the gold settles into the cup through the water, and dry it on the hot plate, the use of the blotter being unnecessary as the cup itself is absorbent. Anneal over a Bunsen or in the muffle, put the cups into the box and take to the balance for the weighing of the gold.

The loss of gold in parting and the incomplete removal of silver from it can cause large errors in gold assays by careless work. The annealed gold must be of a pure golden color on all sides: a piece of pure parted gold will be kept in the balance case for comparison.

Tabulate your weights and results under these headings:

Date	Assay No.	Sample No.	Wt. Bullion	Wt. Gold	Au Fineness.

ASSIGNMENT 6. ALLOYING OF GOLD AND SILVER BEADS.

EXPERIMENT.—Compare the color of 56 with that of 51, 52 and 53. Attempt to part a small piece (about 5 mgs.) of 56 in the same way as 51, 52 and 53 were parted; then anneal and compare the color with that of pure gold. Record results.

As you have seen, when an alloy of gold and silver contains at least 2½ parts of silver to one part of gold, or not more than 28% of gold, the silver can all be removed by proper treatment with nitric acid. When, however, this proportion of gold has been passed, it is found that the insoluble gold protects the soluble silver from the action of the acid so that only a part of the silver will be dissolved. In order, therefore, to obtain the gold in such an alloy in a pure state, it is necessary to alloy with it before parting such an amount of silver as shall make it consist of at least 2½ parts of silver to one part of gold. It is true that there is no ratio of the two metals at which the silver, from being completely removed, suddenly ceases to be removed at all; for in general, with less even than two parts of silver to one part of gold, a portion of the silver will be dissolved, the amount depending upon the strength and temperature of the acid, upon the duration of the boiling and upon the thickness of the alloy; but for its certain and complete removal, as an analytical operation in ore assays, the alloy must contain at least 2½ parts of silver to each part of gold.

A gold-silver alloy containing less than about 40% of gold, or less than 400 fine in gold, has the color of pure silver; above this percentage of gold, the color passes through tints of yellowish-white and brassy yellow to the color of pure gold; but the alloy still has a distinctly brassy cast, as seen when compared with pure gold, even when it contains 98% gold and 2% silver. In this connection, compare carefully the colors of the set of gold-silver test beads which will be found in the balance room. Hence it cannot be told whether a white dore bead contains 40% of gold or whether it contains 28% of gold, (the greatest percentage which any alloy can contain and still part completely), or whether it contains any gold at all. In such a case, direct trial must be made (using only one of the beads, if duplicate assays have been made) to see if it will part, by treating the flattened bead with the acid. It is well to add the weak acid first; if that fails to act on heating, pour it off and add the dilute acid. If the alloy is acted upon by the acid, as shown by its losing its metallic luster and by its changing to a brown-

ish black, amorphous mass, with evolution of oxide of nitrogen, the parting should be completed and the color of the annealed residue compared with that of pure gold, to see if all of the silver has been removed; but if the alloy remains white and metallic or appears to be acted upon only partially, pour off the acid, wash, dry and alloy the bead and repart.

Weigh off two 5 mg. pieces of each of the three samples 54, 55 and 56, cut within a few tenths of a milligram and then weighed to .01 mg. as before. Estimate as nearly as you can by means of the test beads the fineness of each bullion in gold and record your estimate. Weigh off within ½ mg. the following amounts of test silver:

Test silver : Sample 54 :: 2½ : 1
Test silver : Sample 55 :: 2 : 1
Test silver : Sample 56 :: 1 : 1

With one set, fuse the bullion and the silver into a globule with a blowpipe on a smooth cupel which has previously been well ignited in the muffle, keeping the globule fused for a few seconds after it has rounded up to ensure a thorough mixing of the metals. Do not heat the bead too strongly, else a slight spattering may occur. Unite the bullion and silver of the duplicate set of assays by "brightening" them on hot cupels in the muffle with just enough lead to wrap the assay up in. The alloyed beads are then to be flattened on a clean anvil to reduce them to the thickness of an ordinary card and parted as was done in the last assignment, using at first the 1.16 acid, then the 1. 26 acid.

Compare the color of your annealed gold on all sides with that of parted gold known to be pure. If in doubt at any time as to the purity of a gold residue, alloy it with three times its weight of silver and re-part, with especial care to avoid mechanical loss. If properly parted at first, it should weigh the same as before to .01 mg.

In the above work, we have added 2½ times its weight of silver to alloy a bead of a deep yellow, twice its weight to a bead of a light brassy yellow and once its weight to a white bead which would not part, aiming to have the alloyed bead safely contain 2½ times its weight of silver and actually making alloys of 2½ to 4 times as much silver as gold. This procedure is advantageous for the reason that the gold will surely remain in one piece and should ordinarily be followed, the

amount of silver (a reagent) added being, however, guessed at and not weighed. But it has the disadvantage that there is little margin for errors in guessing the weight of silver added: if we add too little, the gold will not be freed from the silver; if we add too much, the gold will break up, using 1.16 acid at first. To obviate these difficulties, we may add a large excess of silver, say ten times the weight of the bead, and then part slowly with 1.05 acid at first, as with sample 53.

If the gold only is to be determined in a sample, an excess of test silver (as above) can be put into the button before or while cupelling: this has the advantage of diluting the gold in the bead with silver on the cupel, preventing, or at least thus minimizing, any accidental mechanical loss which might occur there with a very small bead, as well as doing away with the necessity of alloying with the blowpipe.

A neat way in which to measure the amount of alloying silver used without weighing it (described to me by Mr. Norris English,) is as follows: Make up an alloy of test lead and test silver, by melting them together, containing about 0.100 silver and 0.900 lead; roll this out to such a thickness that each disc cut out by a small leather punch will weigh 10 mg. and contain 1 mg. of silver. One of these discs can be hammered into the lead button before cupelling for each milligram of alloying silver desired, and thus silver determined as well as gold without the necessity of fusing with the blowpipe, the silver added in this way being, of cause, subtracted from the weight of the dore bead; or, one disc can be cupelled with the already cupelled and weighed dore bead on an ignited cupel by means of the oxidizing flame of the blowpipe for each milligram of alloying silver found to be necessary from the color of the dore bead and its weight. The weight of silver per disc can be determined accurately by cupelling 8 or 10 of them and weighing the resulting bead.

Except in cities, and hence at most gold mines, gas is not available and other sources of heat must be used. A convenient arrangement is to use a small kerosene stove to heat a sandbath (pie-plate filled with tailings) on which the capsules are placed; the annealing is done over an alcohol lamp with a flame of good size, thin capsules being chosen in order that the heat may readily penetrate them. Another method is to remove one side of a box of suitable size and replace the top

with a sheet of card asbestos which is perforated with a dozen or two holes to receive the capsules; the alcohol lamp is then moved around under them with the hand to keep the acid at the proper heat during parting, and annealing is done in the same apparatus with the alcohol lamp: this way has the advantage of cleanliness, simplicity and a ready control of the heat. A third way is to put a piece of heavy sheet iron or a sand-bath on top of the portable coke furnace or the gasoline crucible furnace and to do the annealing in the muffle; but the heat of the plate is not readily controlled and dirt is extremely liable to fall into the capsule during annealing in the muffle. Either the first or the second method is better than the third.

References on Parting.

1. Furman's "Manual of Assaying", page 131.

2. "Losses of Gold and Silver in the Fire Assay", Furman's "Assaying." Appendix.

3. Brown's "Manual of Assaying", pp. 224-228.

4. Bodemann & Kerl's "Assaying", pp. 196-200.

Answer the following questions in your notes:—

1. Describe as fully as you can the phenomena seen in annealing: at what lowest heat does the change occur? is the change of color instantaneous or gradual? what other changes occur besides that of color? what is the physical explanation of these changes? For an explanation of this change, see Louis' "Handbook of Gold Milling". An account of investigations on this subject by the same author will be found in Trans. vol. xxiv. pp. 132 and p. 705.

2. Calculate a) what percentage of its own weight of silver must be added to a bullion 0.560 in gold, 0.440 in silver to make the resulting alloy contain exactly 2½ times as much silver as gold. b.) to a bullion containing 0.700 gold, 0.300 silver. c.) to a bullion containing 0 850 gold, 0.150 silver.

ASSIGNMENT 7. EXPERIMENTS ON CUPELLATION AND PARTING.

In the preceding pages, the purpose has been to describe the procedure in cupellation and parting which experience has shown to yield the best results when they are used as analytical processes. The purpose of the experiments to be outlined is to give a wider view of these operations and to determine the losses of silver and the retention of lead and copper in the bead during cupellation under various conditions.

EXPERIMENT 1.—Error by "driving" the assay too hot.

Prepare three assays, each assay in triplicate, one about 100 mg. test silver and 15 gms. lead; another 10 mg. test silver and 15 gms. lead; the third 1 mg. test silver and 15 gms. lead. Cupel one of these sets in the front of the muffle, feathering the cupels carefully as usual; and at the same time cupel a duplicate set in the extreme rear of the muffle, without using coolers: note the color of the back of the muffle. After finishing these six, cupel the third set at the back of the muffle with the muffle at a scorifying heat (a full yellow) so that the cupels and fumes are white. Take care that all beads are brightened at about the same heat.

Calcuate, in each case, the number of ounces per ton of silver which would be reported if each button came from the scorification of 1-10 A. T. of ore. Also, calculate the percentage of the actual amount of silver present which is obtained in the bead in each case.

EXPERIMENT 2.—Effect of "freezing" a cupellation.

Prepare one set (of three assays) as in expt. 1 and allow them to freeze slowly toward the end of the cupellation; remelt them, brighten well and weigh. Calculate results as before.

EXPERIMENT 3.—Effect of the Presence of Copper in the assay.

Repeat a part or the whole of the assays under expt. 1, except add 250 mgs. copper to each assay. Calculate the results as in expt. 1.

EXPERIMENT 4.—Effect of an Excess of Lead on the Silver Results.

Repeat a part or the whole of the assays under expt. 1, except use only ½ gm. of lead in each case. Calculate the results as in expt. 1.

EXPERIMENT 5.—Retention of Copper in the Bead.

Determine the least amount of lead necessary to free an alloy consisting of 400 mg. cupelled silver, 100 mg. copper, from

copper by cupelling first with 2 gms. of lead and increasing the amount by 1 gm. until the bead rounds up. See Aaron's "Assaying", Part 11. Study the appearance of the bead and test a portion of each bead for copper. Determine the copper in the well-shaped bead by the color of an ammoniacal solution. Do not allow the solution to become diluted in making these tests.

EXPERIMENT 6.—Cupel a weighed amount of test-silver (about 500 mgs.) with lead and brighten cold. Weigh the bead, study its appearance and test it for lead with sulphuric acid, after precipitating the silver with hydrochloric acid.

EXPERIMENT 7.—Cupellation and Parting of Platinum.

Cupel together 100 mg. platinum and 5 grams of lead. Also cupel together duplicate assays, each 100 mg. platinum, 200 mg. silver. Treat one with nitric, the other with sulphuric acid.

EXPERIMENT 8.—Parting of Alloys Very Poor in Gold.

Make (from sample 53 and test-silver) one alloy containing 1 part gold: 5000 parts silver, another alloy 1 part gold: 10,000 parts silver and part to get the gold in one piece.

EXPERIMENT 9.—Use of Lead as an Alloying Metal.

Alloy a weighed amount (about 10 mg.) of parted gold on charcoal in the reducing flame with six times its weight of lead, treat the alloy with nitric acid and weigh the washed gold.

EXPERIMENT 10.—Use of Copper as an Alloying Metal.

Same as 10, but substitute copper for lead.

EXPERIMENT 11.—Use of Mercury as an Alloying Metal.

Amalgamate a weighed amount (about 10 mg.) of parted gold with a small drop of mercury and part with nitric acid; wash and weigh the gold.

What are the advantages and what the disadvantages of silver, lead, copper and mercury as alloying metals?

EXPERIMENT 12.—Effect of Chlorides in the Parting Acid.

Part duplicate assays (each about 25 mg.) of 51 in 1.16 acid as usual: pour off the dilute acid and, without washing, add the 1.26 acid and 1 drop of dilute hydrochloric acid to each capsule; heat the strong acid, pour off and wash with water as usual. Now wash out the silver chloride from one of the capsules with strong, hot ammonia, then with water; do not wash the other with ammonia; anneal both. Compare the results with the correct assay.

Does chlorine in the acid dissolve gold under these conditions?

Is silver chloride liable to be weighed as gold?

ASSIGNMENT 8. MELTING, REFINING AND SAMPLING OF GOLD AND SILVER BULLION.

Read before beginning the assignment:—

1. Furman's "Assaying", Appendix A, page 371.
2. Aaron's "Assaying", Part II, pages 21-29.

The purposes of melting bullion before assaying it are (1.) to obtain an average sample through making the whole lot thoroughly homogeneous by fusion. (2) to produce from several pieces or a mass of retorted amalgam, nuggets and gold dust or precipitated gold from a chlorination works one bar of clean metal which can be shipped without chance of some of it being lost or pilfered. (3) to avoid fraud.

The purpose of refining before assaying is to remove dirt and elements which render the bar brittle and non-homogeneous, as lead, zinc, sulphur, tellurium, etc.; it does not pay to attempt to remove copper or silver from gold on the small scale.

Bullion may be cleaned from a comparatively small amount of dirt and dross by putting a few spoonsful of borax crystals into the graphite pot with the bullion at the time of melting to form the bar and skimming off the slag just before pouring: this is all that is necessary with most bullion. Mill amalgam should be cleaned before retorting by picking out iron with a magnet, diluting with mercury and straining, as described in Louis' "Handbook of Gold Milling", page 384. Gold dust should be panned fairly free from sand, dried, and the magnetite and sand blown away before melting. In the case of base or impure bullion, a dip or stream sample should be taken.

In the melting room of one assay office where gold and silver deposits are purchased, chip samples are relied upon only for gold bars of pure bullion (such as those from melting placer gold); stream samples are taken of all silver bars and of gold bars where arsenic, lead, zinc or any other element tending to make the bar non-uniform is present, and of all large gold bars. In taking this stream sample, the melt is first well stirred, sometimes for 2 to 3 minutes; during the pouring, one or two samples of the molten metal are taken from the stream as it runs and poured into small pans of water to granulate them. Thus a correct sample of the melt is obtained and a tedious and expensive refining in the crucible avoided, as the small lots are afterward put together and refined by cupellation with lead on the large scale, the purpose here being merely to sample and value them separately by assay.

Weigh the bar plus the grains which you receive from the one who melted it before you, expressing its weight in ounces and hundredths of an ounce.

Melt it down in the annealed graphite pot with a few spoonsful of borax crystals, adding a little sand to imitate the dirt generally present before melting. When the bullion is thoroughly melted at a low yellow heat and the slag is liquid, remove the slag by means of the skimmer, thus:

Touch the skimmer to the slag, which will chill it and cause it to adhere to the skimmer. Press the slag on the plate to flatten and chill it. Repeat this operation until nearly all the slag is removed, allowing the slag to remain on the skimmer so that no metal may adhere to it.

After removing the slag, the bullion shows a mirror-like surface when first skimmed. This will soon be covered with a scum of copper oxide, to reduce which, cover the surface with a layer of powdered charcoal. Should the bullion not be sufficiently refined at the first skimming, put on more borax but not enough to cover the surface of the melt, allow it to melt on the surface and the base metals in the melt to scorify off and skim again.

While the bar is being melted, the mould is to be warmed and smoked over a luminous flame and a stirrer heated to redness in the furnace. Now stir the molten bullion in the crucible and pour it into the mould which is placed (in this instance) in a miner's pan, a helper taking a stream sample as the bullion runs, for the sake of comparison of the two methods, covering its surface immediately with powdered charcoal.

Obtain the samples for assay by cutting a little more than 1 to 2 grams from each of two diagonally opposite corners, one on top and the other at the bottom of the bar. Also, dry and hammer out the granulations of the dip sample on a polished anvil and weigh out from it a little more than 2½ grams for an assay sample. Assay in duplicate by cupellation, as described in assignment 4, both the chip sample and the stream sample, thus checking both the sampling and the assaying; also assay the samples by Gay Lussac's volumetric method.

"Clean up" carefully, so as to make the losses of silver during melting as small as possible by scraping any globules which may have been left sticking to the walls of the pot from it and by crushing and panning the slag if it is possible that it contains any "shots". Add to the chipped bar these and any other cleanings and the granulations of the stream sample not needed and weigh them all together.

REPORT OF RESULTS OF MELTING AND ASSAYING.

Bar Before Melting.

Weight	xx.xx ounces
Fineness Stated	o.xxx
Calculated weight of fine silver received	xx.xx ounces

Bar After Melting.

	Sample A	Proof Centre	Sample B
Bullion	500		500
Silver		476 (say)	
Copper		24	
Lead	2500	2500	2500
Cupelled Bead	xxx.x	xxx.x	xxx.x
Cupellation Loss		x.x	
Silver Fineness	o.xxx		o.xxx
Fineness by Gay-Lussac Assay		o.xxx	

(Of course, this form will be needed for both the chip and the stream samples.)

Weight of chipped bar, grains, etc.	xx.xx oz.
Weight of chips	.xx oz.
Total weight of bullion returned	xx.xx oz.
Total weight of fine silver returned	xx.xx
Loss of fine silver in melting	o.xx oz.
Weight of fine silver passed to next man	xx.xx ounces

MELTING OF THE COPPER BAR.

The purpose of this work is to give practice in melting, pouring and casting a quantity of metal equal to that in a brick of gold of average size. The melting point of copper is nearly that of gold.

Get a solid bed of coke in the furnace and surround the annealed No. 8 graphite pot, with its bottom resting 2" to 3" above the grate bars on fine coke, with the fuel. Place the copper bar in the pot and melt down by a strong heat. Smoke the large mould over a luminous flame and heat it over the fire. When the melt is thoughly liquid and at a bright orange or a full yellow, such that on casting it will make a smooth bar, lift the crucible from the fire with the clasp tongs and pour it promptly and without interruption but with a steady hand into the mould.

As soon as the metal solidifies, turn the bar out of the mould and immerse it in a tank of water or of dilute sulphuric acid

to remove scale and dirt; scour it with sand and smooth off any roughness of the surface with a hammer.

1. Weigh the copper bar in Avoirdupois pounds and ounces.
2. Convert this weight to ounces and hundredths of an ounce Troy.
3. Calculate the weight and value of a bar of pure gold of equal size, the sp. gr. of gold being 19.3 and of copper 8.8.

REFINING, MELTING AND TOUGHENING OF PRECIPITATED GOLD OBTAINED IN A CHLORINATION WORKS.

The U. S. Mints receive all bullion which is over 0.500 in fine gold, paying coin value for the gold and the market price for the silver; from this are deducted charges for melting, assaying and refining, the charges for refining varying with the grade of bullion. No charge is made for refining bullion over 0.990 in gold and which is entirely free from lead, so that it will pass certain bending tests for toughness and malleability; since then, according to the coinage laws, it is ready to be melted down with copper to form coin gold, which is 0.900 gold, 0.100 copper. A schedule of these charges may be obtained on application at the Mint.

Bars from amalgam and gold dust are never refined at the mine on account of the proportion of silver in California gold; it does not pay, even, to attempt to remove all of the copper on account of the expense and risk of loss in refining small lots, but precipitated gold is so nearly fine that it pays to purify it until it will pass the mint tests. The following notes were made on the method used in carrying this out at the Gold Bank Mine, Butte Co., by the kindness of Mr. H. P. Stow, manager, and Mr. Herrin, assayer and foreman of the chlorination works.

The gold precipitate cleaned up from the vats is heated to the boiling point by means of steam in an earthenware jar with dilute sulphuric acid for about an hour, and is then filtered out on heavy filtering paper and washed until free from acid, the purpose being to remove iron. After draining, it is put into the melting pot by means of a scoop, without drying. The filters are washed off in a gold precipitation vat and all filtrates thrown into the copper vats.

This washed, moist gold, in the condition of a fine, brownish-black powder, is put a little at a time, as fast as that before added melts down, into a graphite pot in the fire along with

crude borax and a little sand to protect the pot. The ash-pit door is closed and the flue stopped with a closely-fitting fire-brick to cut off the draft, while more of the precipitate is being added, in order that none of it may be blown away. A stirrer made of a piece of an old graphite pot is heated in the crucible while the gold is melting down. The gold should remain in the furnace long enough to become thoroughly melted before skimming, so that no globules may be attached to the slag. It took with a rather large melt about two hours to get the melt hot enough to skim. After skimming, the surface of the melt is partially covered again with borax and re-skimmed after a time, this being repeated until the surface is almost clear, there being very little movement on it. The stirrer is used not only to stir the melt just before pouring (which is done, although such fine bullion is sure to be homogenous) but also to feel about in the melt before skimming to be sure there are no lumps in the bottom of the pot but that all is thoroughly fused.

After the surface has been made tolerably clear and free from movement by repeated additions of borax, about a tablespoonful of sal ammoniac (ammonium chloride) is thrown on top of the bullion but not stirred in. Of course, some of it converts the lead into chloride, which volatilizes together with the rest of the sal ammoniac. The addition of sal ammoniac, a tablespoonful at a time, is continued until the surface of the metal is clear and *there is no more movement.* The melt is now apparently free from base metals, especially lead; but to finish the *toughening* of the bar by removing the last traces of lead, a tablespoonful of corrosive sublimate (bi-chloride of mercury) is thrown on top and allowed to volatilize, this being repeated as many times as may be thought necessary. The melt is then stirred, poured and the bar sampled by chipping in the usual way, the chipped corners being rounded off with a hammer. The bars are 0.993 to 0.996 gold, the rest being silver.

Slags and worn out pots from bullion melting are saved until there is quite a quantity on hand, when they are crushed and worked up with mercury in the mill clean-up barrel.

Retorted amalgam is simply melted with borax and skimmed repeatedly until the surface is almost clear, in the usual way. It is poured without oil in the mould, a little oil being squirted from an oil can onto the surface of the bar after casting. The bars so produced are bright and clean and 0.975 to 0.985 in gold and silver together.

ASSIGNMENT 9. THE SAMPLING OF ORES AND METALLURGICAL PRODUCTS.

References.—

1. Furman's "Manual of Assaying", Chap. II.

2. The Sampling of Ores without the Use of Machinery. Wm. Glenn. Trans. Am. Inst. Min. Eng.

3. Brown's "Manual of Assaying," pp. 185-194.

4. The Theory and Practice of Ore Sampling. D. W. Brunton. Trans. xxv.-826.

5. Machinery for Ore Sampling. Bridgman. Trans. xxii. 656.

6. The Sampling and Measurement of Ore Bodies in Mine Examinations. Edmund B. Kirby. 13th Report Cal. State Mineralogist.

Read at least the first *two* references *before* beginning work.

An assay sample must possess the two following qualities:

1. It must be a correct average of the whole lot of ore which it is meant to represent; or, in other words, the following proportion must hold:

Weight of Whole Lot : Weight of Assay Sample : :

Weight of Gold or Silver in Whole Lot : Weight of Gold or Silver in Assay Sample.

It is evident that this proportion must hold true of the small amount of ore put into the scorifier or crucible as well as of the whole assay sample.

2. The assay sample must be homogeneous as regards the distribution of gold and silver throughout its mass. This is accomplished by crushing the small amount of ore selected for the assay sample to pass a fine screen (of 80, 100 or 120 meshes to the linear inch), so as to reduce each particle of gold or silver or of a gold or silver mineral to a very small size, and by then thoroughly mixing the resulting powder. Notice, however, that this gives only a close approximation, although the best one attainable, to the absolute homogeneity found in gases, liquids, solutions or molten metals which have been stirred or shaken.

The richer the ore in the metal which makes it valuable, the less is the ratio of the weight of one particle of the rich mineral to the weight of all the valuable metal in the sample, and hence the less the percentage error introduced into the sample by the presence of one particle more or less than the number which will make the sample of correct, average content.

An iron ore is generally valueless if it contain less than 30% iron; smelters will, generally, not pay for less than 5% of copper or lead in a gold or silver ore and average mercury and tin ores contain 1% to 3% of these metals; while an average silver ore contains only 1-10% of silver (30 ounces per ton) and an average gold ore contains only 1-1000% (3-10 of an ounce, or $6 per ton). From these considerations it follows that silver and, especially, gold ores must be many times more difficult to sample correctly within a certain percentage of the amount present than an ore of the base metals.

There is a wide difference between the natural homogeneity of gold and silver ores from different deposits and localities, or between different ores, with respect to the evenness with which the gold and silver content is distributed, depending upon the gold and silver and other minerals present and the way in which they are associated. For example: Silver sulphide commonly occurs in ores evenly distributed through and evenly mingled with a many times greater weight of sulphide of lead which, itself easily pulverized and diluting, as it were, the sulphide of silver, renders it easy in this case to get a homogeneous assay sample. On the other hand, gold commonly occurs as metallic grains or crystals of varying coarseness which, being rich in gold (50% to 95%) and resisting pulverization on account of their malleability, render it impossible to get a *perfectly* homogeneous assay sample and a matter requiring care and labor to get a satisfactory approximation to it, where the gold in the original lot was quite coarse.

A table of the relative homogeousness of various ores and products may be drawn up as follows:—

HOMOGENEOUS.

Chemical precipitates from leaching works.

Lead-silver ores.

Tailings from amalgamation.

Sulphides concentrated from amalgamated pulp.

Ores in which the gold is present in fine particles, so that, although it yields to amalgamation, it shows no "colors" on panning.

Ores in general containing large percentages of auriferous sulphides but some visible gold.

Ores containing true silver or gold minerals, such as cerargyrite and tellurides of gold and silver.

Mill products containing amalgam.

Scale ores containing gold or silver in metallic particles of such a size as to be readily visible on panning.

NON-HOMOGENEOUS.

Gold ores are more often difficult to get a satisfactory sample of than silver ores, both because gold occurs commonly in the metallic state while silver is commonly combined in minerals and because, gold being more than 30 times more valuable than silver weight for weight, the *content* of gold ores in ounces is less than 1-30 that of silver ores for the same *value* in dollars. Moreover, the extreme accuracy of the gold assay brings it about that difficulties or errors in sampling, not in assaying, often limit the accuracy attainable in valuing a lot of gold ore.

Since amalgamation removes all coarse gold, the pulp in a gold mill after passing the plates, the sulphides concentrated from it or the final tailings are quite homogeneous, so that duplicate assays of any of these should agree closely, provided that the amalgamation has been done carefully and skillfully.

From the above considerations, it follows that a low grade gold ore containing scales offers the most difficulty to the preparation of a homogeneous sample which is, within the limit of accuracy desired, an average of the whole lot.

The obtaining of the assay sample may be divided into two stages:

1. The obtaining of the gross sample, weighing from a few ounces to several tons and obtained by one of the methods described by Furman, according to circumstances. The gross sample is generally taken by the mine foreman or by the superintendent at a mine or by the ore samplers at a smelting works, not by the assayer, and hence we need not consider here how it is done; see references 1 and 6 above.

2. The working down of the gross sample to the small amount put into the crucible or scorifier. What follows describes in detail methods of doing this.

Accurate sampling depends upon the proper performance of three operations: 1. Sufficiently fine breaking or grinding of a sufficient amount of ore. 2. Thorough mixing of the ore after breaking or grinding it. 3. Impartial selection of the sample from this mixture of crushed ore.

1. Breaking or Grinding.

The fineness to which ore must be crushed to give results

reliable to within 1% has been very carefully investigated by Brunton as recorded in the paper quoted above: the fineness for any particular sample may be obtained from the diagrams which he gives. As much ore as specified is, however, rarely crushed except where grinding machinery is at hand. Specific directions for ordinary work where only hand tools are available will be found below.

The most satisfactory hand tools are a large mortar mounted on a block which goes into the earth and a rather heavy pestle with a long handle, for coarse breaking of ore down to about 10-mesh size; and a flat cast iron rubbing plate about two feet square having low edges at the sides, with a 40-lb. muller and, perhaps, also a lighter one, for fine grinding. Whatever tools are used, they should be capable of being easily cleaned.

The meshes of the screens, particularly of the 80 and 100-mesh, should be evenly spaced: whether they are so can be readily told by holding the screen up to the light, and this test should be applied when purchasing a fine screen.

2. Methods of mixing.

a) By Coning. The ore is shovelled into the form of a cone by placing each shovelful squarely on the apex of the cone or by emptying the ore from sack or bottle so that the pieces roll down equally on all sides, taking care to "keep the apex vertically above the point of its first beginning." This method is very commonly used where a large gross sample is being reduced by quartering.

b) By rolling on canvas. Performed by lifting the corners of the sample cloth on which the ore lies. The hand should be held low, so that the ore does not slide down the cloth but rolls over on itself. It is an effective method where the sample is of medium size and coarseness.

c) By passing through a screen considerably coarser than the largest particle in the sample. This method is quite effective with fairly fine pulp when repeated several times.

d) By plowing furrows with the fingers in the sample as it lies spread out on a sample cloth. Effective with a sample finer than 50-mesh. An alternation of b and d in mixing a sample is rapid and effective.

Methods of mixing can be studied experimentally by mixing together soda and pulverized charcoal, or any two powders of contrasted colors.

3. Methods of Selecting the Sample.

a) By Quartering. As a result of much experience of many men on a large variety of ores, this is generally considered the most reliable method. It may always be applied with safety, but is slow. Coarse pieces from the sample kept should not be allowed to roll down into the quarters rejected. Theoretically, the ore should be crushed and mixed between each quartering; but this is rarely done.

b) By the use of sampling troughs, or "assayer's riffles." An excellent sample may be obtained by letting a stream of ore flow onto the troughs from a funnel which is passed back and forth over them. If the troughs in width are ⅓, ¼ or any fraction of the space between separate troughs, the sample is quickly, easily and accurately reduced. This method employs identically the same principle as do the best automatic sampling machines, that of taking the whole of a falling stream of pulp for a fraction of the time.

c) By taking dips over the surface of the ore spread out in a thin layer, the same method which you are accustomed to use in weighing off from the assay sample. Very rapid, and reliable after proper mixing of the sample for homogeneous material, such as vanner concentrates, tailings from amalgamation and some silver ores.

d) When it is desired to have determinations made on the same sample by more than one chemist as a check on one another's work, the following method should be used: Prepare a proper assay sample,—representative, finely ground and thoroughly mixed. Place a number of sample bottles equal to the number of samples desired on a cloth or in a pan, put the ore into a funnel with not too large a spout and, while the ore is running from the spout, pass the funnel around and around over the bottles.

SPECIFIC DIRECTIONS FOR WORK.

It is well before crushing the sample to save a small, representative piece for a specimen.

Unless the sample received by the assayer, weighing probably from 1 to 50 pounds, is already broken to pass a 4-mesh screen, it should be broken to pass a 2 or 4-mesh or finer first and then be reduced by quartering. If the original sample weighed 5 pounds or more, the sample ought to be crushed to pass a 30 to 60-mesh screen when it has been quartered down to 1 to 3 pounds, according to the weight of the original sample.

It is then thoroughly mixed and an assay sample of ½lb. taken out by quartering, the use of riffles or dipping, which is ground to pass a 100-mesh screen (taking care that all scales pass the 30 to 60 and the 100-mesh screens) and thoroughly mixed.

Dried mill pulp and tailings samples are apt to contain clots, which are easily gotten rid of by sifting the sample on a 20-mesh screen and then mashing up the clots on the screen. As the sample contains no coarse gold, it is simply spread out on the sample cloth and ½lb. taken out with a spatula by dips over the whole surface. The asaay sample is ground to pass a 60 or 80-mesh screen.

The above description of methods of sampling supposes the sampler to have at hand only a mortar, rubbing plate, sample cloth, screens and spatula; the method will give a good sample if the work is well done. Sometimes, however, the making of so many samples is required in a limited time that it is physically impossible for the sampler to break up as much ore as that above stated, in which case the sample will have to be quartered to a smaller size before re-crushing or coarser screens will have to be used; but the sample will not be so reliable. The amount of labor to be put upon any sample depends upon the care with which the gross sample appears to have been taken, and the lack of uniformity with which the values in the sample are supposed to be distributed.

When the sample submitted weighs less than one pound, it all should be passed through the 100-mesh screen.

Finely crushed material in sacks, such as vanner concentrates, is accurately and quickly sampled without removing from the sacks, especially if slightly damp, by the use of the sampling spoon.

At a California smelting works, where very careful sampling is done, the whole of the gross sample (weighing from a few hundred pounds to several tons) is first passed through a mill rock breaker and reduced in size so that it will pass a one-inch mesh, then quartered by hand to about 75 pounds. This 75 ponnd sample is broken to pass a 4-mesh screen in a small laboratory ore breaker and quartered down to about 15 pounds. The 15 pounds are ground in a sample grinder to pass a 100-mesh screen, the sample mixed and reduced until of a size to fill the sample bottles. Of rich ore or of ore in which the values are not uniformly distributed, duplicate samples are often made. Of course, it is practically impossibile to make such accurate samples in large numbers except where machinery is at hand for the breaking and grinding, nor is it necessary except where a lot of rather rich ore is to be bought and sold on the assay of the sample made.

Preparation of the Charge.

The following methods of selecting from the assay sample the portion weighed for each scorifier, of measuring the fluxes, of weighing off the ore and of mixing the charge are to be followed with all samples of which you make scorification assays.

Correct methods of sampling should have been carefully followed in preparing the assay sample from the original lot; it is equally important that the assay sample be thoroughly mixed before weighing off and that the small portion put into the scorifier or crucible be an average of the assay sample. Empty the ore onto a sheet of glazed paper and spread it out to a thin layer with the spatula. If you have any doubt as to its being properly mixed, it should be thoroughly mixed before weighing off. Take dips from at least nine spots grouped symmetrically over the sample in getting the portion weighed on the scale pan, taking care, with each dip, to drive the spatula to the bottom of the ore.

Shot measures are provided for measuring the granulated lead and small glass tubes graduated with a paper label in grams for measuring borax glass. Set the shot measure so that it will deliver 30 grams of lead.

In weighing off, spread the sample on glazed paper in front of the pulp balance, place the assay ton weights on the left-hand pan, the ore on the right-hand, hold the spatula in the right hand and turn the balance key with the left hand. The balance should be turned out of action each time ore is put on or off the scale pan and the pointer need move only 1 or 2 divisions to indicate whether too much or too little has been put on, thus making more rapid the operation of weighing.

Pour about one-half the lead required into the scorifier, add the ore, mix with the spatula, spread the rest of the lead over the charge and sprinkle the borax glass on top of all.

Sample Lead–Silver Ore, a smelting ore valuable for both its silver and lead content.

Prepare three charges of this sample, each as follows:—

Ore	1-10 Assay Ton
Granulated Lead	30 grams. Use 2¼ scorifiers.
Borax Glass	½ gram

This charge is one commonly used for silver ores, as it will treat correctly any lead-silver ore, whatever the proportion of galena or of cerussite or anglesite and gangue, and even though

it contain considerable amounts of other more difficultly decomposed sulphides, such as pyrite and zinc blende; while a button of convenient size for cupellation remains. It is, in fact, a charge of universal application for silver ores, whatever the gangue, the amount of galena or other metallic sulphides, or whatever the silver minerals present, with these limitations and modifications:

a) If the ore contains more than 50% of pyrite or of any mineral rich in copper, the lead must be increased in proportion, up to 45 grams for pure pyrite.

b) If the ore contains much lime, zinc or antimony, the borax glass must be increased to 1, 2 or 3 grams to convert these refractory oxides into a fusible slag.

c) If the sample be at the same time homogeneous and very rich, the weight of sample taken may be decreased to 1-20 A. T., thus facilitating the rapid and complete action of the lead and of the litharge formed from it.

Sample Milling silver ore (or a "dry ore"), valuable only for its silver and gold content.

Prepare two charges of this sample the same in all respects as that used for the sample of lead ore above. Prepare a third charge with the same amounts of lead and borax but with only 1-20 A. T. of ore.

Wash 1-10 A. T. of each sample on a batea, making yourself familiar with the minerals present in this condition; notice their relative amounts. Also wash 1-5 gram on a watch glass, as recommended by Furman.

The two samples typify the two principal classes of silver ores, which are in strong contrast from a metallurgical standpoint: the first, containing lead in valuable quantity, would have its lead and silver extracted by smelting; the second would, under usual conditions, be treated by amalgamation or by leaching, perhaps after having been given a chloridizing roast.

Make all six of the scorifications at one time in one of the small muffles. Place the scorifiers as far back in the muffle as possible and close the muffle door. When the lead has melted the door is opened to admit air for oxidizing the ore and the lead. It is quite important that the draft holes at the back of the muffle should be clear so that there will be a strong current of air in the muffle when the flue damper is open. The muffle should be at a bright red before the charges are put in and the heat should be maintained at an orange (or a little above a

good heat for cupelling) during the roasting and scorifying; towards the end, the heat should be increased to a full yellow (not a bright yellow). If the muffle is too hot while the roasting is going on, spurting is liable to occur. Notice the formation of the "bull's eye" as the oxidation progresses. Pour the assays at a full yellow into the warm scorification mould, holding the scorifier in the warmed tongs, as soon as the slag has covered over the lead. Pour carefully into the centre of the cup of the mould, else lead, splashing to the side of the cup, will solidify there and the button, instead of being in one solid piece, will have prills attached, which may be overlooked and lost in cleaning the button from slag.

Describe in your notes the appearance of the scorifier, slag and button after the assay has been poured; also break a scorifier and compare it with the cupel and compare the shape of the inside of the scorifiers before and after use.

Cupel two buttons of the second sample (one from 1-10 and one from 1-20 A. T. of ore) and one button of the first in the same row three inches from the front of the muffle, as usual, feathering them well; for the sake of noting the increased cupellation losses when the cupels are not feathered, cupel the other three buttons at the same time in one row at the extreme back of the muffle, using no colors; thus all six cupellations will be made at one time. Make a diagram in your notes showing, by means of the assay numbers, the position of each assay in the muffle. The cupels which are to be used should be put in as soon as the assays are poured from the scorifiers, in order that they may be ignited while you are slagging the buttons. Do not put in the leads until the muffle has cooled to a proper heat for cupellation.

Weigh each dore bead separately; part the three beads from each sample together in one capsule and weigh the gold from the three assays of each sample together.

Example of Form of Record of an Ore Assay:

Date	Assay No.	Sample No.	Ore	Dore	Gold	Silver	Oz. Au	Oz. Ag	Gold Assay Value	Remarks
	1	9	1-10							
	2	34	1-10							
	3	34	1-20							

ASSIGNMENT 11. SCORIFICATION ASSAY OF QUARTZ-PYRITE GOLD ORES.

The silver in an ore can be determined with quite as much accuracy as is needful by taking 1-10 A. T. for each assay, since we thus determine the content of the ore to 1-10 of an ounce or its value to six cents a ton, assuming the sample perfectly homogeneous and no inaccuracy in manipulation. When, however, we determine the gold to 1-10 of an ounce by this same method, we have determined its value to only $2 a ton, which is not sufficiently precise with any except unusually rich ores. We must, therefore, treat more ore for each assay and thus obtain a larger amount of parted gold for weighing, so that each hundredth of a milligram represents a smaller value per ton. As we can never take more than 1-5 A. T. for each scorifier of ordinary size and shape and only 1-10 A. T. of ores consisting chiefly of metallic sulphides (other than galena), we are generally obliged, after taking as much ore in each scorifier as possible, to run several scorifications and unite them by re-scorifying the leads together or by cupelling the buttons separately, parting and weighing the cupelled beads together. No more ore than can be perfectly scorified (1-10 to 1-5 A. T.), as shown by a clear slag, clean scorifier and bright lead, must, however, be taken for each scorifier.

Moreover, as assay samples of gold ores cannot be made in many cases, when containing coarse "colors" of gold, as homogeneous as would be desirable, it is advisable to treat as much ore for each assay as can be conveniently done in order that our result may give the average of a larger amount.

a) Quartz Gold Ores Containing 0 to 50% of Pyrite.

Sample 50. Quartz with coarse gold and a large percentage of pyrite and other sulphides.

Sample 24. Concentrations from a "canvas plant" in Butte Co., Calif. The pulp from which this sample was concentrated had previously been amalgamated in the battery and concentrated on vanners, so that the sample contains no coarse gold. It contains about 50% of pyrite.

The assay samples furnished of both ores have been put through a 100-mesh screen and thoroughly mixed so as to render the resulting fine pulp of the assay sample as homogeneous as possible.

Weigh out 1-10 A. T. of each sample and wash on a batea. Make yourself familiar with the appearance on the

batea of the minerals in the samples, noticing, moreover, what proportion in mass as seen on the batea is formed by the percentage said to be present by weight. Free gold can hardly be found by washing in a sample so finely ground. The same tests may be made with 1-5 gram of sample on a clock glass.

Samples such as those above can be assayed correctly by the use of the charge given for the silver ores; the other substitute charges given, equally correct, permit the use of a larger amount of ore in each scorifier. That method will be chosen for the assay of these ores by scorification by which the largest amount of ore can be treated with least time and labor.

Follow the method of sampling the ore and of mixing the charge that was used in assignment 10. Run one of each of these three charges with both samples, thus making six in all, at one time in one of the small muffles:

	1	2	3
Ore	1-10 A. T.	1-6 or 0.15 A. T.	1-5 A. T.
Gran. Lead	30 grams	45 grams	50 grams
Borax Glass	½ "	½ "	½ "
Scorifier	2¼ inch	2½ inch	2½ inch

The scorification of sulphide ores in general can be divided conveniently into five periods:—

1. Melting. Put the charges as far back in the muffle as possible at a low yellow and close the muffle door. If the fire is right, by the time the charges have melted thoroughly the muffle will be reduced to an orange heat by the introduction of the cold scorifiers. It is important that the melting be thoroughly done, else when the muffle door is opened to admit air for roasting, sulphur di-oxide gas, escaping through the viscous surface of the charge, is apt to cause spurting and mechanical loss.

2. Roasting. Maintain an orange heat with a good air current in the muffle, so that the reactions proceed briskly and yet the charges do not sputter. Should they begin to spit, close the muffle door until the surface of the charge has melted more thoroughly or draw them toward the front of the muffle to lessen the heat. Notice the roasting or burning of the pyrite by direct action of the air when the charges are first put in, as shown by its glowing, exactly the same as on the hearth of a reverberatory roasting furnace or as would occur in

the scorifier if no lead were present. The sole purpose of reducing the heat at this period is to prevent mechanical loss. Open the top doors of the furnace, so as to cut off the draft through the muffle, and smell of the fumes given off from the charges, as they rise from the mouth of the muffle.

3. Fusion, or formation of the "bull's eye." When the tops of the charges have become quite smooth, showing that most of the sulphur has been roasted off, close the muffle door and increase the heat to cause the contents of the scorifier to fuse down thoroughly, until a good "bull's eye" forms, as with the silver ores. It is often well to place large, hot pieces of coke in the front of the muffle during this period. Notice that a much higher heat is needed to form the "bull's eye" with quartz ores containing pyrite than with galena. It is important for a good scorification to bring about the formation of the "bull's eye" as early in the process as possible and to thoroughly fuse the contents of the scorifier at this time. Notice that when the heat is raised a more or less vigorous effervescence in the slag begins, showing that chemical reaction is taking place in it, resulting in the evolution of a gas. Up to this point in the scorification, the sulphur, arsenic, antimony and base metals have been oxidized in large measure directly by the oxygen of the air; now, on raising the heat, the litharge reacts with the remnant of sulphur and other oxidizable substances in the charge, burns them completely and a perfectly fused slag results.

4. Scorification. Having the charge thoroughly fused, open the muffle door, close the dampers except the flue draft, so as to maintain a strong draft through the muffle and a moderate heat, and allow the lead to scorify or slag off until the ring nearly or quite closes. By the scorifying, not only is the amount of lead to be cupelled reduced, but lead oxide is added to the slag, rendering it still more fluid and more strongly oxidizing.

5. Final Heating. Increase the heat to full yellow just before pouring (if not already at this heat), in order to give the scorifier and its contents such an excess of heat that the slag will remain thorougly fluid after withdrawal from the muffle while the assay is being poured.

Weigh and record the weight of each lead button before cupelling it, this once, in order to study the advantages of the various charges. Reduce the size of any buttons over 20 grams in weight before cupellation by re-scorification: replace the

button, after knocking off most of the slag, in a scorifier already heated in the muffle and allow the "bull's eye" to cover over again.

Cupel, part and weigh the dorè and the gold separately for each of the six buttons. Average the results of the three assays of each of the above two samples for the number of ounces per ton and the gold assay value, after calculating them separately for each assay. It will give a good check on the weighing to weigh all the gold from each sample together, after first weighing each prill separately.

Answer the following questions in your notes on finishing the work to this point.

1. Show by calculation within what limit in dollars and cents the gold assay value of an ore can be determined when the gold is weighed to .01 mg. and 1-5 A. T. of the sample is taken for assay; when ⅓ A. T. is taken; 1 A. T.; 5 A. T.

2. Which charge of the three above given do you consider preferable for the assay by scorification of quartz-pyrite free gold ores? Give a full discussion, based upon the data as to weights of lead obtained, time required for scorification and amount of ore worked, as you found them before the furnace in carrying out the assays of these samples with each charge.

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b) Scorification Assay of Vanner Concentrates; and of Ores Containing over 50% of Pyrite.

Sample 23. Concentrates made on Frue vanners at a forty-stamp mill of Butte Co., Calif.

About nine-tenths of the sample is pyrite and this mineral decides the assay treatment; the other minerals are in too small an amount, in this sample as well as in the vanner concentrates of California in general, to have any influence whatever on the choice of the assay method, their presence being detected only by qualitative tests in the wet way. Vanner concentrates from the mines of California often contain besides 60% to 90% of pyrite and some gangue, small percentages of chalcopyrite, arsenopyrite, zinc blende, galena, etc.

This sample contains no free gold, as the pulp from which these concentrates were obtained had been carefully amalgamated by experienced men before passing onto the vanners. Where the amalgamation has been poorly done, the concentrates often contain amalgam, which causes the sample to give slightly varying results on assay.

Weigh six charges of the sample, each charge as follows:—

Concentrates	1-10 A. T.	
Granulated Lead	45 grams	Use 2½ inch scorifiers.
Borax Glass	½ gram	

Make all the scorifications at once in one of the small muffles and manage the furnace in the same way as was done in scorifying the pyrite ores in 11 a); refer back to that. On account of the large amount of sulphur present, take especial care to obtain a good "bull's eye" as early in the process as possible, by supplying plenty of air to the charges during the roasting through a good draft in the muffle and by raising the heat from orange to a full yellow to form the "bull's eye" just as soon as the charges are fairly smooth on their surface. Notice that the charge given above is the same as in charge 1 in 11 a), except that the lead has been increased to 45 grams. Only 1-10 A. T. of concentrates can be treated in a 2½ inch scorifier.

For the scorification assay of pyrite concentrates in general, it is well to run six charges, each of 1-10 A. T. of the sample, with lead and borax as above and unite the buttons in threes by re-scorification; then if the gold is weighed to .01 mg., we have duplicate assays, each of which should give the gold to .03 or .04 of an ounce, or inside of $.75 per ton, which is as close as is generally required for concentrates.

Unite three of the leads from the scorification of sample 23 by re-scorification before cupelling; i. e., replace three of the leads in a new scorifier after knocking off most of the slag and allow to scorify until the slag again nearly or quite covers over the lead. It saves time to put the scorifier into the muffle to heat as soon as the scorifications are poured and while the buttons are being cleaned. Cupel the other three buttons directly and in separate cupels; weigh each of these beads separately for the dorè; part one of them alone, the two others together. The carrying out of the work as described will give three results, which should check, and which illustrate different methods of working.

The advantage of uniting the buttons before cupellation is that when the sample contains three or four times as much gold as silver, so that the dore beads will be very small, a very small loss on the cupel from a 1-10 A. T. bead would represent not only a large percentage loss, but a large loss in value per ton. But this loss, should any occur, will be lessened proportionally by cupelling together the lead from a larger amount of ore. With small beads, rich in gold, it is important that the bead

should be seen to rest on a smooth spot on the cupel. When the approximate content of the sample is known, and little silver is present, the alloying silver (sometimes eight or ten times as much as the gold present) is often added to the lead button before cupelling; and this practice is to be recommended as the silver serves to collect the gold into one bead on the cupel.

REFERENCES ON SCORIFICATION IN GENERAL.

1. Furman's "Manual of Practical Assaying", pp. 122-126.
2. Bodemann & Kerl's "Assaying", pp. 107-116. This gives an excellent account of scorification and should be carefully studied.
3. Brown's "Assaying", pp. 105-214 in the 5th edition.
4. Kerl's "Assayer's Manual", pp. 129-133. Especially, has a valuable table on page 131.
5. Mitchell's "Manual of Practical Assaying", pp. 611-617 in the 6th edition, edited by Crookes.

ASSIGNMENT 12. SCORIFICATION ASSAY OF SAMPLES RICH IN COPPER.

The principal difficulty in assaying for gold and silver material containing a considerable percentage of copper lies in getting a lead button for cupellation sufficiently free from copper, so that the silver can be entirely freed from it in cupellation and so that the cupellation losses will not be excessive. If the buttons obtained from scorification are hard and, especially, if they have a more or less distinct copper-red color in spots, they need to be re-scorified (with the addition of more lead if the buttons are small) to a fairly pure and malleable state before cupelling.

EXPERIMENTS.

Before making the scorifications, carry out the following experiments:—

Expt. 1. Weigh on the pulp scale and brighten on a hot cupel considerably above a feathering heat:

Test silver 250 mg. Copper 30 mg. Sheet Lead ½ gram.

Expt. 2. The same, except use 2 grams of lead.

Expt. 3. The same as expt. 2, except omit the copper. This is of course for the sake of comparison.

Describe fully in your notes, for each experiment, the appearance of the assay while cupelling and of the bead after it has finished; give an explanation of what is seen.

Flatten the beads from experiments 1 and 2, cut off one-half of each, dissolve in nitric acid and test for copper with ammonia; re-brighten what remains of each bead on a hot cupel with 1 gram of lead. Describe results fully.

From the above experiments, it is evident that the lead buttons, in addition to being scorified as free from copper as possible, should be of good size (at least 10 grams). Feather the cupels during the first part of the cupellation, but increase the heat gradually for the last three or four minutes and have it at a bright red for a half minute during which the cupellation is finishing: all of which is, of course, to favor the elimination of copper from the bead. Should the bead still come out impure, it may be re-brightened on a hot cupel with ½ gram of lead; but this will cause a slightly increased loss of silver and a lower result on ores containing an important amount of silver.

Sample Copper Precipitate from a Chlorination Works, Butte Co. Rich in finely divided gold.

Sample 278. A high grade Copper Matte.

Determine gold as well as silver in both samples; most of the difficulty in assaying is, however, generally in the silver.

Prepare two charges of each sample exactly like that for vanner concentrates. Prepare one charge of each sample the same, except use only 1-20 A. T.

Manage the furnace heats as in assignment 11. Put the charges of 278 in the back of the muffle in scorifying, on account of the larger amount of sulphur which it contains.

Cupel the three buttons from each sample separately, on the same line in the muffle, working very carefully and according to the directions above. Describe the cupellation of each assay accurately in your notes, as the results depend very largely upon how the cupellation is done.

Part all three buttons from the assays of sample 278, after weighing each bead separately for the silver, together in one capsule. Part the beads from each assay of the other sample separately.

REFERENCES ON THE ASSAY OF CUPRIFEROUS SAMPLES.

1. Furman's "Assaying", page 250.
2. A Uniform Method for the Assay of Copper Materials for Gold and Silver. Ledoux. Trans. xxiv–570.
3. Assays of Copper and Copper Matte. Trans. xxv–250.

Assignments 10, 11 and 12 are intended to describe in detail the best methods of making the scorification assay of three contrasted and the three most common and important types of ores and products which present themselves for assay by this method. Almost all ores fall easily under one of the three types; exceptionally an ore will require a slight modification of one of the three methods given; e. g., an ore containing a large proportion of antimonite or zinc blende may be assayed as a silver ore, but the borax glass needs to be increased to one, two or three grams and a bright yellow heat applied to bring about complete fusion in the scorifier. Refer for these exceptional cases to the table given in Furman's "Manual."

Notice that one of the chief advantages of the scorification method is its ready application, with only occasional slight modifications, to all sorts of materials; its most serious disadvantage is that the amount of ore needed for the sufficiently precise determination of the gold cannot be treated in one scorifier.

ASSIGNMENT 13. PRACTICE ON MISCELLANEOUS SAMPLES.

Assayers are required to assay from four to fifty samples a day, having at hand, of course, for the larger amounts of work large muffles and the best of apparatus. To accomplish this without sacrifice of accuracy, an assayer must not only possess skill, which comes with practice, and have at hand apparatus corresponding to the amount of work to be done, but he must carry the assays through the various operations of scorification or fusion, cupellation, parting and weighing in as large batches as can be handled conveniently with the apparatus available and must adhere to some such system as that outlined in the preliminary suggestions (refer back to pages 5 and 6) and in what follows.

The purpose of this assignment is to give you practice in applying the methods previously learned to a variety of ores,

to increase your skill and power of independent work and, especially, to accustom you to a system and order of work by which a large amount of work can be done in a limited time without sacrifice of accuracy. As this assignment is intended to develop speed, the furnace work on the number of samples given *must be completed* during the day; do them as well as you can in this time but *get them done anyway*. You have before this worked solely to attain accuracy; now, for the sake of practice, work to attain reasonable dispatch, putting accuracy second at the start and you will, if possessed of fair skill in manipulation, soon find yourself able to combine the two. A concentration of the mind on the work, combined with the use of sufficient and the right kind of apparatus and proper method, will enable you to get a large amount of work done without impairing the accuracy of results.

This assignment is, in effect, a test of the skill which you possess in manipulation, as judged by the amount of work done in the given time and the accuracy of results on unknown ores. You will receive the prepared assay samples in the morning, will have a furnace for your exclusive use and will be expected to have completed the cupellation of and the weighing up of the beads during the day.

The assay samples, as soon as received, should first be arranged in some convenient order, preferably that of the sample numbers, and the sample numbers recorded opposite the assay numbers in tabular form similar to this:

Assay No.	Sample No.	Marked	Mineral Composition	Type No. A. T. Ore	Wt. Doré	Wt. Au	Wt. Ag	Oz. Au	Oz. Ag

Next determine the mineral composition by washing 1-10 A. T. of each sample on a batea so as to separate the different minerals according to their specific gravities without washing any of the sample off the batea. Separate by washing and determine with the blow-pipe any mineral which you do not

recognize by simple inspection. Estimate the percentage of each mineral as closely as you can and record the percentage composition of each sample in the fourth column of the above table. It may be well to write on only every other line in the table and to record the mark on the sample and the mineral description under the sample numbers on the next pages of the notebook.

You should now be in a position to refer each sample to one of these five types: 1. Silver or silver-lead ore. 2. Gold ore with more than 50% of quartz. 3. Gold ore with more than 50% of pyrite. 4. Ore with a large percentage of copper. 5. An exceptional ore, requiring a modification of one of these methods. Consider whether the ore is likely to be valuable chiefly for its gold or its silver; since, as was said before, one should take as much ore as he conveniently can in assaying a gold ore, while the result for silver will be obtained quite as accurately from 1-10 A. T. as from more. Record the type number in the fifth column. Decide on the amount of ore to be used and write the amount of ore in A. Ts. in the sixth column. A convenient system for doing a lot of samples is to use always a 2¼ inch scorifier and 1-10 A. T. of ore; 30 to 45 grams of lead (according to whether much or little pyrite is present), and ½ gram of borax glass, unless the presence of large amounts of lime, magnesia, zinc or antimony render necessary the use of more borax glass. With unusually rich samples which are uniform, at least one check should be run using only 1-20 A. T. Make at least two assays of each sample and more where you think it needful.

Now write on a board of about the same shape and size as the floor of your muffle day numbers in the order of the writing on a page, cover each number with a scorifier and weigh off your charges. Take to the furnace, reversing the order from front to back in the muffle, but letting it be the same after pouring into the mould as on the board. As soon as the scorification is finished, at once put in the cupels, so that they will be ignited by the time you have cleaned the buttons. Let the order in the cupels be the same as on the board. To avoid mixing samples, never vary the order and *handle only one assay at a time*; mark scorifiers and cupels with the day numbers, if you prefer.

To accomplish most, keep your muffle full—12 scorifiers in the 12x8x5 muffle, 24 cupels; 6 scorifiers, or even 8, in each

12x6x4 muffle, 12 cupels. Finish the sampling of all the ores before beginning the furnace work of any, then the furnace work of all before beginning the parting and weighing of any. Weigh out the second batch while the first muffle full is scorifying.

Fill out the remaining columns of the above table at the balance as the weights are obtained, and make the calculations to ounces as soon as you have finished weighing.

ASSIGNMENT 14. CRUCIBLE ASSAY OF QUARTZ ORES.

The samples of this class are generally either mine or mill samples and comprise:

1. Ordinary ores sent to the mill, or the mill feed; or the pulp after it has passed the battery.

2. Samples taken from pits or trenches on the outcrop of a deposit to determine whether it is worth working.

3. Samples taken from any part of the workings of a mine to determine whether there will be a profit in stoping and working the ores they represent.

4. Samples taken from the breast of a drift or the bottom of a shaft or winze (even where it is known that the ore is below workable grade) to determine whether the shaft, drift or winze is approaching or running out of an ore chute or body of workable ore.

In the last three cases, the sample may prove to have any content from a trace to a moderate value.

The importance of accurate assays of such samples to enable the mine to be worked most profitably, especially where, from the character of the ore, its value cannot be known approximately by panning, by its appearance as judged by one very familiar with that particular deposit, or by any other way than by assays, can readily be comprehended. Inasmuch as low grade ore, where it can be worked at all, occurs in large bodies, each sample, either from the mine or mill, may represent a large amount of ore and hence its value per ton needs to be determined with some precision: the total error of sampling and assaying should not exceed at most fifty cents per ton. This class comprises the majority of samples assayed at many gold mines, the only other samples being those of bullion, concentrates and tailings.

The preparation of an average and homogeneous assay sample of an ore in which the gold is free and occurs in rather large particles is a matter requiring care and considerable labor;

there are no appreciable sources of error in the assaying for gold of a homogeneous sample, where the work is skillfully and carefully done: in fact, the method is probably one of the most accurate analytical processes known. There are, of course, numerous errors which can be made with unskillful or careless work.

Sampling.

The gross sample of the mill feed is obtained by taking a little ore from the lip of the feeder of each battery every hour, half-hour or oftener. These portions are thrown into a pile and quartered down together. The pulp is sampled by passing a cup through the stream every hour or every half-hour or by the use of an automatic sampler run by the shafting. Mine samples are taken as described by Kirby in reference 6 of assignment 9. The gross samples are reduced to the assay sample by the methods described in assignment 9.

Assaying.

These samples are, perhaps, the easiest to assay of any we have. The only error likely to be made in the fusion is in not allowing the charge to become thoroughly melted or in a loss of lead in cleaning the button or by the charge boiling over. The only error likely to be made in the cupellation is that a small bead is liable to be split up into several beads, some of which may be lost, should it rest on a rough spot or in a crack of the cupel. It is not at all necessary to feather the cupels if silver is not to be determined; or even if it is to be determined, since the silver is generally of small content and value. The only errors which need be guarded against if good materials and reasonable care are used lie in a splitting up of the bead on the cupel, in retention of silver in the parted gold, loss of gold in parting and inaccurate weighing, all of which you know how to guard against.

If gold only is to be determined in samples which contain little silver, the alloying silver can be put into the lead button before or while cupelling; in fact, it is advisable to do this, not only because it renders the operation of alloying the bead unnecessary, but also because the gold is then collected in a larger bead on the cupel.

DIRECTIONS FOR WORK.

Sample Copy the label into your notes. Determine its mineral composition by vanning ½ A. T. on a batea. The sample consists principally of quartz, with coarse, free gold.

It is an example of an ore of no reducing power. It has been ground to pass a 100-mesh screen and then thoroughly mixed.

Prepare two charges, each:

Ore	1 A. T.		
$NaHCO_3$	50 grams	or Na_2CO_3	35 grams
Litharge	50 "		
Argol	1½ "	or 1 gm. of flour, or 0.6 gm. of charcoal.	

Mix and cover with 10 grams of ground borax.

The only trouble in the assay of ores of this type lies in the difficulty of obtaining an assay sample which shall be both fairly homogeneous and a correct representative as to its gold content of the whole lot of ore from which it was taken. In order to obtain a reliable result, correct methods of sampling must be conscientiously followed and the assay sample must be crushed to pass an 80 or preferably a 100-mesh screen, taking care that all metallic particles which cannot be proved with a magnet to be iron pass through the screen. The sample should be very thoroughly mixed after sifting and the *mean* of 2 to 6 assays reported as the result.

Sample Copy the label and van the sample, as above. The ore is valuable for its silver as well as its gold; the gold is fine, so that duplicates should agree closely. Prepare one charge the same as that given for the last sample.

Use F crucibles for all three of the above charges and fuse all in the top of the small muffle furnace at one time.

Prepare one charge of this sample as follows in a 12-gram muffle crucible and fuse in the muffle with those of the next sample:

Ore	½	A. T.
Na_2CO_3	10	grams
Litharge	50	"
Argol		as needed.

Mix and cover with 10 grams of ground borax.

Sample Copy the label and van the sample, as above. The sample contains 10% to 15% of pyrite. Run two charges in the muffle like the second charge for the last sample.

In mixing the charges, put the soda into the crucible first (in order that no ore may remain unacted upon at the bottom of the crucible), then the litharge and other fluxes, lastly the ore and mix all together with a fork, sprinkling the cover of ground borax (hydrous) or salt over the top.

Fuse the three charges in F crucibles in the fire on top of the muffle at the same time the charges in muffle crucibles are being fused in the muffle.

Cupel all of the buttons obtained in this assignment at one time in the muffle. Determine both gold and silver in all. As the second contains the most silver, cupel these buttons in the front row, feathering the cupels.

Heat the charges at first at such a moderate red that the bubbling shall go on not too briskly. When they are quiet, increase the heat to low yellow, as shown by the color of the inside of the crucibles, for about five minutes, until the tops of the charges having borax covers show that the fusion is complete by becoming smooth on top, like the surface of a well melted scorification. Pour into the crucible mould. Save the buttons to be cupelled with all the others of this assignment.

The slag of a properly fluxed and thoroughly fused charge on pouring will be of thin creamy consistency and free from shots. If it is too acid, due to not adding enough basic flux to combine with the silica, it will form a thread in pouring, or will even not flow at all. If pasty or lumpy, it has not been heated long enough. If not thoroughly oxidized, there was not enough litharge in the charge.

The crucible, after the charge has been poured, should be lined with only a thin glaze of the slag and should be little corroded. There should be no particles of undecomposed ore sticking to the sides of the crucible nor should any lead "shots" be seen in it after pouring, while still hot.

The button should weigh 10 to 20 grams, be bright and soft and separate readily from the slag.

The slag ought to be homogenous and perfectly oxidized; it should be transparent, glassy and so brittle as to crack to pieces on cooling. The last three properties are not necessary for a correct assay, but are properties of slags which separate well from the button and which give evidence that the ore has been perfectly decomposed. If the slag is too acid, especially if it does not contain enough litharge, it is apt to be tough and tenaceous and to adhere strongly to the button, making it difficult to separate them. If 65 grams of litharge and 35 of soda are used instead of 50 of each, as above, the button will separate cleaner from the slag.

If the cups of the mould are dipped into cold water just after the red has left the slag, the latter will be rendered very brittle by the sudden chilling, so that often it will break

off perfectly clean from the button all around at one blow of the hammer; but water must not be allowed to get into the cups, or the mould will be rusted and it will be rendered harder rather than easier to clean the buttons.

The assay ton system is the simplest which could be devised where Troy ounces of gold or silver are to be reported per short ton of ore. With low grade gold ores, however, it is the common custom to express their value not in ounces, but by their gold assay value in dollars and cents; as "Three Dollar Ore", "Ten Dollar Ore", "the tailings assay forty cents". Now the gold assay value can be obtained just as readily and with as little calculation if the ore is weighed in grams as in assay tons; for, (refer to page 7) if 100 grams of ore be taken for assay, each .01 mg. of parted gold indicates a value of $.06 per ton. Where 10, 25 or 50, instead of 100, grams of sample are used, each .01 mg. indicates a value, of course, of 60, 24 or 12 cents per ton instead. In metallurgical tests in the laboratory, such as roasting, leaching, amalgamation and concentration tests, it is convenient to use the metric system, and it will simplify calculation if the ore for assaying, also, be weighed in grams instead of in assay tons. Of course we can obtain by a simple calculation either the assay value when the ore is weighed in assay tons or the contents in ounces when the ore is weighed in grams, should it be desired. The above system for the use of gram weights instead of A. T. weights in certain cases and the method of calculating the gold assay value was originated by Professor Christy.

FUSION IN THE MUFFLE. USE OF A MIXED FLUX.

Make one assay of and one of , using the quartz flux which you will find in the bin and the special form of crucible with a broad bottom and low sides, known as muffle or Colorado crucibles, which are intended for making fusions in large muffles with straight sides. Make the fusion of the blast furnace slag, as put down in assignment 15, at the same time in the straight side muffle. It is told by the sound when the bubbling has ceased; then the assays are heated to a full yellow for five or ten minutes longer. Cupel the buttons with all the rest from this assignment.

The quartz flux was prepared as follows: equal amounts of litharge and sodium bi-carbonate were weighed out and to them argol was added in the proportion of 1½ grams to each 100 grams of the litharge and soda together, or enough to produce

15 grams of lead, and the ingredients were thoroughly mixed together on a sample cloth. A tin box was then cut off at such a length that it would hold when full 100 grams of the flux, which is the amount required to flux 1 A. T. of a quartz ore. This gives exactly the same charge as was used with these ores before. In using the flux, put a measure full into the crucible, place the weighed ore on top, stir up with a fork or spatula and cover with about ten grams of ground borax.

Whenever one has often to assay samples which are about the same in composition each time they occur, as e. g., concentrates from vanners produced by the same mine, it saves work to make up a suitable flux and provide a measure of the right size for that class of samples. Eg. We may prepare another flux mixture and another measure for a charge like the second one given for the Comstock ore, for silver ore and samples rich in gold and ores with 5% to 15% of pyrite; and another flux mixture and measure for concentrates.

Any quartz ore containing not more than 5% of sulphides and not too much lime or magnesia will give a perfect slag with these charges, so that modifications are only occasionally required. The one A. T. charge answers well if a small amount of sulphide is present; when the sample contains 5% to 10% of pyrite, the ½ A. T. charge should be used, as also with silver ores and perhaps rich gold ores. If no sulphides, or only a small amount of them, are present, the litharge may be decreased, even to only the amount necessary to furnish lead for the button; but larger crucibles will then be required, the effect of the litharge in fluxing the quartz being supplied by more soda. The slag is the more strongly hydroscopic on exposure to the air the more soda it contains, sodium silicate being hydroscopic, lead silicate not. The slags from the 1 A. T. charges above are slightly hydroscopic and hence should be cleaned up from the anvil and slag table at once: if 65 grams of litharge and only 35 of soda were used, the slag could be exposed to the air indefinitely without becoming in the least moist, just like scorification slags.

If much lime or magnesia is present, the borax should be increased to the amount necessary to give a transparent slag, which, in the case of pure serpentine (hydrous silicate of magnesia), is a weight of borax glass equal to that of the ore. Ground borax (hydrous) can be used in the charge up to about 25 grams; in the rare cases in which more is required, borax glass or dried borax should be substituted for it, remembering

that hydrous borax contains only about half its weight of $Na_2B_4O_7$.

Almost any hydro-carbon will serve to throw down the lead; argol and flour are preferable to charcoal as they are more uniform in reducing power and have a less reducing power for unit volume, hence can be measured more exactly.

Read the chapter in Aaron's "Assaying", Part I, on "Materials", page 35.

ASSIGNMENT 15. ASSAY OF TAILINGS, RESIDUES AND SLAGS.

1. Stamp Mill Tailings and Cyanide Residues.

Sample 25. Tailings from a forty stamp mill of California. Determine only gold.

Tailings samples must be taken from the stream of pulp as it runs to waste from the mill, the best by an automatic sampler connected with clockwork or a small water wheel, or with the shafting; if this is not at hand, then by taking a sample of the stream with a cup every hour. The bucket of muddy water thus obtained daily can be settled quickly by the addition of a spoonful of alum, the water poured off carefully and the sample dried. Screen the sample thus obtained through a ten-mesh sieve to mash up the clots, roll on the sample cloth to mix, spread it out on the sample cloth, sample out about 10 A. T. by dipping over the whole surface, screen the sample on a 60-mesh screen and crush what is too coarse to pass.

Prepare one of each of the followlng charges:

Tailings	3 A. T.	Tailings	100 grams
Litharge	110 grams	Litharge	125 "
Soda	110 "	Soda	125 "
Argol	1½ "	Flour	1 "

Use Battersea H crucibles.

The lead buttons should weigh 15 to 25 grams.

The parted gold of duplicate charges of this sample, and of any tailings sample which does not contain amalgam, should agree to .01 mg. You should therefore be able to report the value of the tailings to within seven cents per ton in gold. Should still greater precision be desired, four, or even five, A. Ts. can be fused at one time in a J crucible, with fluxes in proportion. With tailings, the amount of silver in assayer's litharge together with the little generally present in the tailings themselves is almost always sufficient to ensure the proper

parting of the few hundredths of a milligram of gold obtained even from 3 or 4 A. Ts. of the sample.

When a wind furnace is not at hand and the fusion has to be made in the muffle, run three crucibles for each assay with reducing agent to get a 5 to 8 gram button in each and cupel the three buttons in one cupel.

The above charge is sometimes of use with very low grade samples of ore free from sulphides, such as gold-bearing gravel.

2. Blast Furnace Slag.

Sample of Blast Furnace Slag. Determine both gold and silver. Prepare one charge as follows, using a muffle curcible and making the fusion in the muffle.

Slag	1 A. T.
Litharge	15 grams
Charcoal	0.5 "
Soda	30 "
Borax Glass	10 "
Borax Cover	10 "

Blast furnace slag from the reduction of lead ores is a silicate of calcium and ferrous iron with small and varying amounts of other constituents. Refer to Furman's "Assaying" and Hofman's "Met. of Lead."

Stick one nail into the charge, point down.

3. Blank Charge. The following charge can be used to determine how much silver the litharge contains; and it could also be used to determine whether the fluxes you were using at any time had been "salted" by accident or design:

Run one charge in an H crucible:—

Powdered window or bottle glass	125 grams
Soda	100 "
Litharge	100 "
Charcoal	0.3 "
Borax Cover	10 "

The amount of silver in the litharge could, of course, be determined by simply fusing 100 grams of litharge with a half gram of charcoal, a gram of flour or 1½ grams of argol to reduce a button.

ASSIGNMENT 16. CRUCIBLE ASSAY OF QUARTZ ORES CONTAINING FROM 15% TO 50% OF SULPHIDES.

Sample This is the same sample which you scorified before. See assignment 11.

Litharge Assay. Run one charge:

Ore ½ A. T.	Soda 15 grams
Litharge 100 grams	Quartz Sand 15 grams
Nitre "	Borax crystals in charge 10 gms.

Cover of borax. Use an "F" or a 20-gram crucible.

"Boil down" the charges as before; after the charge is quiet, give a full yellow heat for ten minutes: a longer and hotter final heating is necessary for these ores than for quartz ores. The sand is used to protect the crucible from corrosion by the slag, which, without it, would be highly basic. Glass may be substituted for sand but more of it must be used to produce the same effect. The sand used must be free from gold and should be finely ground.

Nail Assay. Run one charge of the same sample:

Ore	½ A. T.	Borax Cover 15 grams
Soda	45 grams	Use an "F" or a 20 gram cruc.
Litharge	25 "	2 Twenty-penny nails

Stick the nails into the charge after the borax cover has been added, points down. Should the nails first added be eaten up by the charge before fusion is finished, take out the small bits remaining of the old ones and add two or three fresh ones, for an excess of metallic iron is supposed to be present in the charge all of the time. The charge given above is that in Aaron's "Assaying". When little silica is present in the ore, it is well to add 10 to 15 grams of powdered quartz sand.

First fuse down the charge at a low to a bright red, then raise the heat to a full yellow, taking care not to cover the crucible quite closely until the charge has ceased foaming; now cover (if the fusion is made in an open fire) and give a very bright yellow heat for 10 to 15 minutes, continuing the heating until, on removing the nails and rinsing them in the slag, they are seen to be free from adhering globules of lead.

The nail assay is a convenient one for ores containing varying amounts of sulphides, as the lead button obtained is of unvarying, proper weight, no matter what the content of sulphides

in the sample may be. It cannot, however, be used for assaying ores which give a button of speiss with the use of the above charge of fluxes, as the speiss will contain, if the ore be at all rich, important amounts of gold; on other ores, it is reliable for gold but does not give quite as high results as scorification or nitre fusion for silver. On account of the iron sulphide remaining in the slag, it is well to have the lead button weigh 20 to 25 grams.

Sample 24. This is the same sample which you have scorified before. See assignment 11.

Litharge Assay.

Run one charge the same as that used in making the litharge assay of the above sample except use 8 grams of nitre.

Nail Assay. Run one charge the same as that used in making the nail assay of the last sample.

The lead button from both nitre fusion and nail assays should be free from matte, and bright and malleable. The slag of a perfect nitre fusion is clear and glassy and as free from matte as that of a scorification assay. The color is brown black, the shade being the deeper the more iron there is present. The crucible should be lined with a thin glaze of clear slag without any trace of undecomposed ore. The slag of a nail assay is coal black in color; it is sometimes of vitreous, sometimes of submetallic lustre. The matte should not surround the button but should be perfectly diffused through the slag.

ASSIGNMENT 17. CRUCIBLE ASSAY OF PYRITIC CONCENTRATES AND OF ORES CONTAINING OVER 50% OF PYRITE.

There are quite a number of different methods in use for the assay of samples of this class, some assayers preferring one method and some another. Many consider that only the one method which they use is reliable. It is my experience that, with proper carrying out of details, the different methods will give closely agreeing results, in general: two or three exceptions will be noted as we come to them. It is largely a matter of convenience which to choose. We will take a sample of concentrates which you have already scorified (see assignment 12) and assay it by all the other methods in common use,–the nitre assay, the nail assay and by roasting. You will then be able to see how near results by the different methods agree as judged by your own work, so far as it is well done and as regards this sample at least. After each method has been described in detail, a comparison will be drawn between them.

a) The Nitre Assay.

1. Determination of the Reducing Power of the Sample.

One gram of pyrite will throw down about 8½ grams of lead, ½ A. T. of a clean concentrate 115 to 125 grams of lead and ¼ A. T. about 60 grams of lead, providing the sulphide is oxidized by litharge alone. When the sulphide is oxidized by nitre, of course no lead is thrown down; hence by varying the amount of nitre used, we can control the size of the button, at the same time that the ore is completely oxidized. One gram of nitre oxidizes exactly 4 grams of lead.

As we wish to reduce, within a few grams, a 20-gram button and as the reducing power of different samples varies (depending upon the percentage of sulphides present), we need to be able to determine or guess correctly the amount of lead which will be thrown down before making the assay, in order to know how much nitre to add.

Make a preliminary assay to determine the reducing power of the concentrates thus:—

Concentrates	1-20 assay ton
Litharge	75 grams
Salt Cover	Use a 5 or 10 gm. crucible

Have the fire hot when the charge is put in and fuse quickly; remove as soon as the charge is quiet, do not pour. If the crucible remains in the fire much longer than is necessary, it is liable to be eaten through by the litharge. When perfectly cool, break the crucible, clean and weigh the button.

Calculate the weight of nitre which must be used with ½ A. T. of concentrates to produce a 20 gram button; also with ¼ A. T.

As too much time would be required to make a preliminary assay of each sample, it is always better to fuse the assay at once with the amount of charcoal or nitre which it is supposed will give a button of the right weight and correct the amount in a repeated assay, should this prove to be necessary. Samples of clean pyrite concentrates have a quite constant reducing power, 23 to 27 gms. of nitre generally giving a 20 gm. button with ½ A. T. of sample. The eye can estimate from the appearance of an ore in lump the proportion of minerals with a reducing action, after some practice; at first most persons are apt to overestimate their proportion. A more accurate method is to van a little of the assay pulp on a batea or large clock glass.

The following method will enable even an inexperienced

person or one out of practice to estimate the reducing power so accurately that a button of the proper weight will generally be obtained at the first trial:—

Procure a sample of perfectly clean pyrite machine concentrates and a lot of quartz sand or of quartz free from sulphides. Grind each to pass through a No. 60 mesh screen. Prepare 10 mixtures as given in the following table, mix thoroughly and preserve in envelopes, labelling each envelope with the composition and reducing power of the standard which it contains.

Standard	Contains		Gms. of Lead Reduced by	
	Pyrite	Quartz	1 A. T.	½ A. T.
1	2%	98%	5	
2	5	95	12	
3	10	90	25	
4	15	85		19
5	20	80		25
6	30	70		37
7	40	60		50
8	50	50		63
9	70	30		88
10	100	0		125

In use, weigh out ½ A. T. of the pulp to be tested and van carefully on a batea. Weigh out ½ A. T. of the standard which you judge to contain the same proportion of sulphide as the sample and van on another batea. A comparison with the eye of the amount of sulphide will show at once whether the guess was correct or whether another standard is nearer to the ore. Should other sulphides than pyrite be present, remember that galena reduces ⅓ as much, chalcopyrite and zinc blende about ¾ as much lead as pyrite for the same weight. Of course, not all the heavy sulphide mineral is obtained at the first vanning in either case; but the same proportionate amount is obtained with standard and ore.

2. Nitre Assay Using ¼ Assay Ton.

Prepare one charge as follows:

Concentrates	¼ A. T.
Litharge	75 grams
Nitre	to give a 15 to 20 gram button.
Soda	15 grams
Sand	20 grams
Borax Crystals	10 grams

Borax Cover. Use a 20-gram crucible.

3. Nitre Assay Using ½ Assay Ton.

Prepare one charge as follows:

Concentrates	½ A. T.
Litharge	150 grams
Nitre (to give a 20 gram button)	
Soda	15 grams
Sand	30 grams
Borax Crystals	10 grams

Borax Cover. Use an F crucible.

Run these two charges together with the ones by roasting and the nail assay, making four charges in all at one time. Run these two charges and the one of the roasted sample in the way described below; leave the nail assay in the furnace at a high heat for 10 to 15 minutes after the other three have been poured.

The slag of each charge should be clear and vitreous, like a good scorification slag from pyrite concentrations; the button should be soft and bright.

The reactions seem to be more complete with sulphide ores when the fusions are made quite quickly. Although nitre fusions boil over when heated too fast, this mishap is quite as likely to be caused by the crucible being heated unequally,—as at the bottom, while there is a crust at the top. Have therefore a good bed of bright red coke before putting in the crucibles and surround them from top to bottom and on all sides with red hot coke, putting the fusion of roasted ore in the hotter part of the furnace next the flue. Fuse the charges as quickly as you can without danger of their boiling over: when they are quiet, give them a low to full yellow for ten minutes to finish. This final heat is important. As samples of concentrates have their value chiefly in gold and are rich, it is best to get a rather large (20 gram) button.

The nitre assay is equally correct for gold with the assay by roasting, when both are properly made and a good slag is obtained; it is also equally correct with scorification when plenty of litharge is used.

b) The Nail Assay.

Prepare one charge the same as that given in assignment 16, except add 20 grams of sand. It is sometimes well to omit the nails, add 20 grams of nitre, and "boil down" the charge; then add the nails and finish at a high heat as usual. The purpose of this is to volatilize as much sulphur and arsenic as possible and is especially advantageous where a large amount of speiss would otherwise be thrown down.

c) Assay by Roasting.

Prepare a cover for your roasting dish by making a small hole in the bottom at the centre and notches in the edge. Line another roasting dish thoroughly by rubbing a blackboard crayon over its interior.

Weigh out ½ assay ton of the sample into the roasting dish. Place the dish, covered, in the muffle, which is to be heated to *low redness only around the edges and corners.* The concentrates should be spread out in the roasting dish and ridged across, so as to expose as much surface as possible to the air. The gentle heating at first is adopted to avoid a too sudden delivery of sulphur di-oxide, which might cause mechanical loss. After one-half hour, take out the roast, letting it cool a little before removing the cover; then turn the ore over with a spatula, leaving it spread about in the dish in a thin layer and ridged across, as before. Return to the muffle without the cover, if the heat is low as described above. After one-half to one hour, take out and stir again. The heat may be increased to a low red when the roasting is partially complete. Continue this treatment until no odor of sulphur di-oxide is perceived on stirring. The ore has then had an "oxidizing roast." Towards the end, increase the heat to a bright red or low yellow for ten to fifteen minutes to decompose any iron sulphate which may have been formed. The ore has then had a "dead roast."

Describe in your notes the appearance of the roasted pyrite.

References on roasting:—

1. Brown's "Manual of Assaying" pp. 273-278,
2. "Leaching Gold and Silver Ores" by Aaron pp. 15-20, 43-48 and p 99.
3. Prof. Christy's paper in the Trans. Am. Inst. Min. Eng. Volume XIII. on "The Volatility of Gold and Losses in Roasting Gold Ores."

Smelt the roasted concentrates with this charge:—

Concentrates–roasted.

Litharge	50 grams	Use an F Crucible.
Charcoal	½ grams or	After the concentrates have
Argol	3 grams	been transferred to the cruci-
Soda	15 grams	ble, the roasting dish should
Sand	20 grams	be "dry washed" out with a
Borax	10 grams	little soda, which is added to
	Borax Cover.	the charge.

This charge can of course also be used for roasted pyrite from a roasting furnace and for chlorination tailings, by increasing the reducing agent.

Comparison of Methods.

Of the nitre methods, the charge given above for ¼ A. T. is used by many and is, perhaps, preferable where a good balance is available for material containing much sulphur, since thecharge, containing much less nitre and ore and being smaller, is not so likely to boil over in a crucible of the size ordinarily used. Moreover, it is preferable for ores moderately rich or very rich in gold or silver in this respect that the ratio of the weight of the lead button to the weight of precious metal in the charge is twice as great. It has, besides, this considerable advantage that it is easier to estimate correctly the amount of charcoal or nitre to add: since, as only one-half as much ore is used, there will be only one-half as much variance in weight of the lead button reduced from the weight desired for any given variance of the real percentage of sulphides from their estimated percentage than if ½ A. T. of sample be used.

On low grade samples or those containing amalgam, it is perhaps better to use the ½ A. T. charge; which can be relied upon to give the same results as two ¼ A. T charges when the sample contains not over five ounces of gold.

The nail assay is used extensively in Colorado and among the mines of the Mother Lode. If well made, it is reliable for gold in the absence of speiss. But since it requires a higher heat and longer time in the furnace than the nitre and does not give so neat and clean a fusion, the latter method is generally preferred for samples high in sulphur. The advantage lies with the nail assay in samples of a moderate but varying amount of sulphur; but with concentrates there is generally little trouble in guessing the nitre to give a button of the right size.

The roasting method of assaying gives the same results for gold as the others, if well done; but as it takes two to three hours to make the roast and just as long to make the fusion, etc., as the others and as the assayer is exposed to chance of mechanical loss in roasting, it is not to be recommended. Silver losses are likely to take place. It is here described that you may learn the operation of roasting, which often has to be performed in metallurgical tests.

Study carefully Aaron's "Assaying," Part I, pp 35-72.

ASSIGNMENT 18. ASSAY OF SAMPLES VERY RICH IN SILVER WITH THE DETERMINATION OF THE SLAG LOSS AND THE CUPEL ABSORPTION.

References:—

1. The Assay of Silver Sulphides. Furman. Trans. xxv-245. Discussion xxv-898. Furman's "Assaying" page 252.

2. The Inaccuracy of the Commercial Assay for Silver. Stetefeldt. Trans. xxiv. With discussions.

3. Silver Losses in Cupellation. God shall. Trans. xxvi-473.

4. Losses of Gold and Silver in the Fire Assay. Furman. Trans. xxiv-735. Reprinted in Appendix to his "Manual."

5. A Uniform Method for the Assay of Copper Materials for Gold and Silver. Ledoux. Trans. xxiv-575. Discussion xxiv-872.

6. "Assaying." Bodemann & Kerl, pp. 103 to 105.

7. Process of Treating Lixiviation Sulphides. Dewey. Trans. xxvi-258 to 261.

8. The Accuracy of the Commercial Assay for Silver. Dewey. Journal Am. Chemical Society xvi-505 to 516.

9. Notes on the Assay of Rich Silver Ores. E. H. Milier and Chas. H. Fulton. Sch. of Mines Quarterly vxii-160.

Cupel Absorption according to Bodemann & Kerl, (p. 104:)

Contents of Ore in Ounces per Ton.	Absorption of Cupel is per cent of weight of cupelled bead.
10 to 35	4%
38 to 70	3%
75 to 145	2%
150 to 225	1%
225 to 4400	¾%

Calculate the weight in milligrams of 1-20 A. T. Test your 1-20 A. T. weight against your milligram weights on the bead balance. Calculate the deviation of the 1-20 A. T. weight from accuracy in percentage, assuming the milligram weights to be standard. Calculate the error in ounces per ton due to the error in this weight on material assaying 15,000 ounces per ton.

Sample 28. Silver Precipitate from the Gold Bank Chlorination Works, Butte Co., Calif. Precipitated from the

sodium thiosulphate solution with sodium polysulphide. Contains lead sulphide, copper sulphide, over 50% of silver sulphide.

Weigh out, on the bead balance, two charges of each sample: Sample 1-20 A. T., Lead 25 grams, Borax glass ½ gram, 2¼ inch scorifier.

Melt thoroughly at a moderate heat, cutting off the draught and closing the muffle door while doing this to avoid possibility of loss by spitting. Scorify at a low heat (somewhat above that for cupellation) and increase it toward the end. Pour. Save both slags, each charge by itself in its own scorifier, properly labelled.

Make two crucible assays of 28 with this charge:

Sample 1-20 A. T., Litharge 50 grams, Soda 15 grams, Argol ½ gm., 5 grams borax glass. Cover of borax. Use small crucibles, melt in the muffle and pour the charges. Save and label the slags, each in its own crucible.

Cupel in sets of two and *as near to perfection as you can:* the accuracy of results will depend largely on the skill with which the cupellation is done. Label and save the cupels. Part the beads without flattening them at all, using very dilute acid and only warming it gently so that the beads dissolve slowly; when action has ceased, as shown by the shrinking of the gold, pour off the weak and add strong acid; this treatment will leave the gold in one piece, if well executed. Test the *cold* silver nitrate solution from parting for copper with strong ammonia. Compare results by scorification and by fusion. Make a full record of observations and deductions in your notes.

Determination of Silver and Gold in the Slags and Cupels.

Re-melt the slag of each assay in its own crucible or scorifier, cut off the draft through the muffle and add to each a mixture of 5 grams of soda with 1½ grams of argol. Cupel the resulting buttons carefully with feathers.

Break off from each cupel and reject the part not saturated with litharge and grind each separately to pass an 80 mesh screen, cleaning the rubber after grinding with 10 grams of sand, which is added to the charge. Fuse each cupel with this charge:—Soda 25 gms., Borax 25 gms., Argol 1½ gms. Borax Cover. Fuse the charges at a medium yellow; feather the cupels. Notice that no litharge is used, hence no silver can be introduced into the charge by the fluxes.

Record the results in this form:—

FIRST CUPELLATION.

	1 Scor.		2 Scor.		3 Cruc.		4 Cruc.	
Dore								
Gold	mgs.=	oz.	mgs.=	oz.	mgs.=	oz.	mg.=	oz.
Silver	mgs.=	oz.	mg.=	oz.	mgs.=	oz.	mg.=	oz.
SLAGS.								
Dore								
Gold	mg.=	oz.	mg.=	oz.	mg.=	oz.	mg.=	oz.
Silver	mg.=	oz.	mg.=	oz.	mg.=	oz.	mg.=	oz.
CUPELS.								
Dore.								
Gold	mg.=	oz.	mg.=	oz.	mg.=	oz.	mg.=	oz.
Silver	mg.=	oz.	mg.=	oz.	mg.=	oz.	mg.=	oz.

Total Oz. Gold.
Total Oz. Silver.

These last results would be reported as the corrected assay. Corrections for slag and cupel absorption are not allowed, according to business custom at the present time, in the purchase and sale of ores; but they may be allowed in the future.

Oz. Silver by Volhard Titration (p) oz.
Oz. Gold on the filter (p) oz.

Assignment 19. Assay of Telluride Ores.

Although almost all of the gold of Nature (and particularly in California) occurs as metallic gold of varying coarseness of grain or crystallization, either alone or intimately associated with metallic sulphides, there are quite a number of gold-silver-tellurium minerals which, although found only in small amounts at any one place in sufficient amount to be identified, generally in "pockets" in the vein, are quite widely distributed both throughout California, in other regions of the western part of the United States and in other countries. Except in picked ore specimens or in the fine sulphides overflowing from the concentrate boxes (sometimes) in a mill, where the telluride minerals tend to concentrate, they do not occur in appreciable amount so as to make necessary or advisable any change in the usual methods of *assaying*, nor does their correct assay offer unusual difficulties, as was formerly thought.

Inasmuch as tellurium occurs in Nature in true chemical combination with gold and silver in minerals which contain

large percentages of the precious metals, it follows that wherever tellurium is present in sufficient amount to be detected, the ore will be rich and each particle of the telluride mineral will contain an important amount of gold. Hence the necessity that the sample be properly taken and the assay sample be ground through at least a 100-mesh screen and then thoroughly mixed, with the same care used for ores containing coarse gold; still, telluride ores are easier to sample than scale ores, since the telluride minerals are easily crushed on account of their marked brittleness.

The most common tellurium minerals are:

Hessite. Ag_2Te. Te 37%, Ag 63%.

Calaverite. $Au\ Te_2$. Te 55.5%, Au 44.5%.

Petzite. Differs from hessite in that gold replaces much of the silver. Analysis by Genth from Golden Rule Mine, Calaveras Co.: Te 32.68% Ag 41.86 Au 25.60%.

Sylvanite. $(Ag\ Au)\ Te_2$.

These minerals have a hardness of 1.5 to 3.5, are silver-white, steel gray or iron black in color and streak, brittle to sectile, with a metallic lustre and no distinct cleavage. For fuller information, see Dana's "Text book" and "System of Mineralogy."

Tests for Tellurium.

A very delicate test for tellurium in ores where not too much base metal sulphide is present is:

Place a little of the ore in fine powder in a white parting capsule, add a few drops of concentrated sulphuric acid and heat gently until nearly all of the acid has evaporated. If tellurium is present, a pinkish coloration will be seen in the capsule which, on treatment with water, changes to a dirty brown. 25 milligrams of an ore containing 35 oz. of Au and 105 oz. of silver, largely as telluride, gave the test very strongly; 5 milligrams showed the reaction.

With samples containing much sulphur and only a little tellurium: oxidize the ore thoroughly with strong nitric acid, evaporate, expel the nitric with sulphuric acid, and treat with moderately strong hydrochloric acid, when the tellurium will be found in solution as chloride. Tellurium is precipitated as a black powder from this solution by stannous chloride and may be filtered off and tested with sulphuric acid by the above test.

In assaying, only small amounts of ore, not over 1-10 A. T.

for scorification or ¼ A. T. for fusion, should be used in a single charge: this will favor a complete reduction of the gold and silver, and will be sufficient since the ore is in general rich. In crucible fusions, plenty of litharge should be used in the slag, say from 50 to 150 grams for ¼ A. T.

Scorification is usually given the preference over fusion in assaying telluride ores, some saying that telluride ores *must* be scorified. I have found, in experiments on rich telluride ores, that results by scorification and nitre fusion agree closely. If there is any advantage in point of consistency and highness of results, combined with rapidity and ease of execution of the assay, it lies with scorification, but the advantage is slight. Furman says that both scorifications and fusions of telluride ores should be put into the furnace at a rather low temperature, which is gradually raised to the usual high heat at the end.

The following results which I have obtained will show the degree of agreement of results by different methods:—

Sample 267. Quartz with calcite, containing a considerable amount of a telluride mineral (perhaps petzite) and some free gold. From a "pocket" at the Melones Mine, Calaveras Co., Calif. Contains only a very small amount of sulphides.

Scorification charges used:

Sample 267	1-10 A. T.	Sample 267	1-10 A. T.
Gran. Lead	30 grams	Gran. Lead	30 grams
Borax Glass	1 gram	Borax Glass	1 gram
		Litharge Cover	10 grams

Sample 393. Sulphides settled from the water overflowing from the concentrate boxes, Carlisle Mine, Butte Co., Calif. Consists for the most part of pyrite but contains a telluride of gold and silver.

Scorification charges used:

Sample 393	1-10 A. T.	Sample 393	1-10 A. T.
Gran. Lead	45 grams	Gran. Lead	40 grams
Borax Glass	½ gram	Borax Glass	½ gram
		Litharge Cover	10 grams

The charges were put into the furnace at a moderate red, increasing the heat gradually to a full yellow at the end. The lead buttons are apt to contain tellurium and it is necessary to take pains in brightening in order to get a pure bead.

Results obtained:

Sample	Method	Ore	Oz. Au	Oz. Ag	Remarks
267	Scor	1-10	69.4	254.1	30 gms. gran. lead
267	Scor	1-10	68.5	255.3	30 gms. gran. lead with a cover of 10 gms. litharge

267	Fusion	½	69.10	260.40	Bead impure.
267	Fusion	¼	68.60	256.36	Bead well brightened.
393	Scor.	1-10	60.5	318.1	45 gms. lead ½ gm. B.
393	Scor.	1-10	60.9	317.3	45 gms. lead with a cover of 15 gms. litharge.
393	Fusion	¼	59.80	312	

References.

Occurrence and Behavior of Tellurium in Gold Ores, F. C. Smith Trans. xxvi, pages 487-490. Discussion: H. Van F. Furman, xxvi-1106. W. P. Jenney, xxvi-1105.

Assignment 20. Assay of Gold Bullion

Read carefully, *before* performing the assay, the two following references:—

1. "Manual of Assaying" by Furman, pp. 246 to 249 and pp. 311 abc.
2. Aaron's "Assaying," part 2, pp. 30 to 47.

In addition, the following references give valuable data:—

3. The Sampling and Assaying of Gold Bullion, by Cabel Whitehead and Titus Ulke. Describes the method practiced in U. S. Mints. Eng. and Mining Journal, February 12, 1898.
4. "The Metallurgy of Gold" by T. K. Rose, chap. ix. The chapter on the assaying of gold bullion contains many valuable results of investigation on the accuracy of the method made at the Royal Mint at London.
5. Bodemann and Kerl's "Assaying," pp. 177 to 198. This reference contains excellent minute directions for cupellation and parting.
6. Kerl's "Assayer's Manual," pp. 167 to 184.

The fire assay of gold bullion is one of the most accurate determinations of chemical analysis: reports are made to ½ of a point, or .05%, reports of two assayers should agree to within one point and duplicates often agree to less than one-fourth of this amount. Since refined gold bullion is easy to sample and since the assay gives such accurate results if the work is well done, an assayer's skill is often judged by the agreement of his gold bullion assays with those of the mint or with those of the refiner to whom the bullion is sold. This assay controls the gold coinage of all countries, which is the world's standard of value of all commodities.

Gold, silver and dore bullion almost always contain copper, in various proportions with the gold and the silver, often small quantities of iron, lead and zinc, and, rarely, in the gold obtained from the placer mines of some districts, platinum.

The bullion assay is a process of refining and its purpose is to determine how many thousandths (points) each of gold, silver and base metal the bullion contains.

PRELIMINARY ASSAY.

It has been found by experience that in order that all except a trace of base metal shall be removed from the cupelled bead, as shown by its rounding up as the cupellation is finishing, a certain proportion of lead is necessary and that this proportion varies with the fineness of the bullion and according to whether it consists chiefly of gold or of silver. More than this, it is customary to make the ratio of silver to gold in the alloyed bead quite accurately either 2:1, in that case boiling the laminated bead twice in nitric acid of the same strength as our stronger parting acid, or else making the ratio in the alloy 2 ½:1 and boiling the laminated bead once in acid of the strength of our weaker acid (1.16 to 1.20 sp. gr.) and once in acid of the strength of our stronger parting acid (1.26 sp. gr.), in order to ensure a proper separation of the silver from the gold, the retention of as small and constant a surcharge of silver in the parted gold as possible and to prevent the gold from breaking up. In order to follow the method accurately in these two respects, we need to know, therefore, before beginning the assay, the approximate comp sition of the sample to determine the amount of lead and silver to be added.

Although, after considerable practice, the assayer can tell with sufficient precision for the purposes above stated the composition of the bullion by inspection, an approximate determination must be made by one who has not had this practice before beginning the assay. There are several ways of doing this quickly and with sufficient exactness. 1. By weighing 50 to 100 mgms., cupelling, weighing the dore and comparing the color of the bead with those of a set of test beads in which the proportions of gold and silver are known. 2. By making a preliminary assay, as follows:—

Weigh off one assay:

Bullion	90 to 100 milligrams.	Wrap the bullion and the
Test Silver	300 "	silver in the lead and
Test Lead	3,000 "	squeeze into a tight wad.

Place in a HOT cupel and cupel at a higher heat than for a silver bead, taking care to finish at a good heat, with no feather litharge. Weigh and part the bead. Record results in this form:

	Milligrams	Thousandths
Bullion taken	90 to 100	
Cupelled bead	———	
Test silver taken	———	
Bullion plus test silver	———	
Base metal (copper)	———	0.xxx
Gold	———	0.xxx
Silver	———	0.xxx
	Total,	1.000

To correct these results, add 15 thousandths to the silver and subtract the same amount from the base metal.

"The following table, from Mitchell, will give an idea of the proportion of lead required to separate copper from precious metals where the dore alloy is chiefly gold, by cupellation:

Precious Metal in Alloy	Lead Required
0.900	10 parts
0.800	16 "
0.900	22 "
0.600	24 "
0.500 to 0.050	34 "

"For alloys where the precious metal is chiefly silver, about half the above proportions of lead suffice, but in both cases much depends upon the temperature of the muffle and upon other considerations; the guide is the condition of the bead: if it is hard, brittle or coppery, it must be treated with a fresh quantity of lead." The bead must round up and show by the absence of scales of copper oxide or lead oxide on its surface as the cupellation is finishing and while the bead is still fluid that it is free from these metals. Furman gives it as a rule to use 6 grams of lead and ½ gram of sample in cupelling refined gold bullion in all cases, and this rule answers very well as this kind of bullion is almost always over 0.900 fine in gold and silver together. The alloying silver should be added before the refining cupellation is made, because when little silver is present it is difficult to separate copper from gold by cupellation.

Before beginning the strict assay, test your set of weights by balancing the 500 mg. piece against 500 mg. of the small pieces; also the 1000 mg. piece against 1000 mg. of the smaller pieces. Of course, all of the weights ought to be consistent among themselves.

DIRECTIONS FOR CARRYING OUT THE ASSAY.

Use 500 milligrams of bullion for each of the duplicate assays, A and B, cut off so exactly (within .05 mg.) that the amount taken may be considered in calculations to be exactly 500 mgs. Wrap the bullion of each assay in 6 grams of sheet lead together with the amount, approximately, of test silver shown to be necessary by your preliminary assay. The test silver added is to be weighed within 0.10 mg. in order that silver may be determined in the bullion. Make up your proof centre from pure gold, copper and test silver to correspond with 500 mgs. of the bullion plus the amount of test silver necessary to alloy and wrap the metals in 6 grams of sheet lead. To prevent any particles of the assays from sticking high up on the cupels, which they are very apt to do, thus causing serious mechanical loss, squeeze the assays into a tight cube with the bead pliers, holding them over a clean, smooth surface while this is being done that nothing be lost; and place the assays in the centre of well-ignited, HOT cupels, so that the assay will melt down at once into one mass.

Cupel quite hot, i. e., much hotter than for a silver assay, so that no feather litharge forms, taking care to have the furnace at a good heat when the cupellations are finishing and leaving the assays in the furnace until the fluid beads are well brightened. The reason for this high heat is that it is rather difficult to separate copper from a considerable proportion of gold by cupellation; besides, if the bead contains a very little lead from not being well brightened, the alloy will be more or less brittle, so that on rolling the bead to laminate it for parting, the edges of the strip will be rough, occasioning probable mechanical loss through the cracking off of small particles.

Clean and weigh the beads. Hammer each into a small, flat bar with smooth edges on a clean anvil and roll out in the rolling mill to the thickness of an ordinary card. In laminating, anneal the alloy whenever it becomes hard and elastic and before it begins to get rough on the edges; also, take only light passes in the rolls, so as to reduce the thickness gradually. At every setting of the rolls, all the strips should be passed through, especially at the last pass, in order that all the strips may be of exactly the same thickness, as the surcharge is partly dependent upon the thickness of the strips. In the absence of a rolling mill the whole of the flattening can be done with hammer and anvil, but the surcharge will be about a point higher and not so constant as when rolls are used. Anneal again when the

laminating is finished and roll the strip into an open coil or crumble it so that it can both be put into the flask readily and the acid can have free access to all of its surface.

PARTING. Boil the cornets gently with dilute acid of 1:16 sp. gr. until action ceases (10 to 15 minutes), using 1½ ounce of that acid; decant off the spent dilute acid and replace with strong acid (1.26 sp. gr.) BOIL GENTLY with this acid for 15 to 20 minutes. It is necessary to add a bit of blow-pipe charcoal as large as a pea with the strong acid in order to avoid "bumping." Decant off the strong acid and wash the gold in the parting flask three times with hot distilled water. Be careful all through that the parting flasks, which are to be used with annealing cups, and the reagents do not become contaminated with chlorides.

Record your weights and results in this form—

	Sample A	Proof Centre	Sample B
Bullion	500.0		500.0
Pure Gold		xxx.xx	
Test Silver	xxx.x	xxx.x	xxx.x
Copper		xx.	
Sheet Lead	6,000	6,000	5,000
Bead after cupellation	xxxx.x	xxxx.x	xxxx.x
Silver added	xxx.x	xxxx.xx*	xxx.x
Gold and Silver	xxx.x	x.xx	xxx.x
Bullion	500.0		500.0
Gold and Silver	xxx.x		xxx.x
Base metal	xx.x		xx.x
Gold and Silver	xxx.x		xxx.x
Gold after parting	xxx.xx		xxx.xx
Silver	xx.x		xx.x
Cupellation loss (thousandths)	.oxxx		
Surcharge (thousandths)	.oooxx		
Corrected Gold Fineness	0.xxxx		0.xxxx
Corrected Silver Fineness	0.xxxx		0.xxxx
Corrected Base Metal	0.xxxx		0.xxxx
Total,	1.000		1.000

*Plus gold added.

Cupellation Loss is the difference between the weight of the

cupelled bead and the weight of the gold and silver which was actually present in the alloy before cupellation. It is a resultant error and is the algebraic sum of the minus errors due to the absorption of gold and silver by the cupel and to their volatilization and of the plus error due to retention of copper and lead in the cupelled bead. Since the minus errors are greater than the plus error where the work is properly done, the cupellation loss is a negative error.

The *Surcharge* is the difference between the weight of the pure gold which was actually present in the alloy before cupellation and parting and the weight of the parted gold. It is a resultant error and is the algebraic sum of the plus error due to the retention of silver in the parted gold and to the minus errors due to the absorption or volatilization of the gold during cupellation and to its solution in the acid during parting. The retention of silver being more than sufficient, generally, to counterbalance the other errors, the surcharge is, generally, a positive error. The parted cornets are about 0.998 fine gold and the surcharge, in good work, lies between 1-10 and 1-2 a thousandth when rolls are used for the flattening; when a hammer and anvil are used, the cornets will be about 0.997 fine and the surcharge will amount to about one to one and one-half thousandths.

The *Proof Centre* is a synthetic assay made up of pure gold, pure silver and pure copper in the same proportions in which they are contained in the assays, as judged from the preliminary assay, which is submitted to exactly the same processes to which the assays are submitted. Its purpose is to determine the cupellation loss and the surcharge so that they can be corrected for. In actual work, proof centres are seldom used to determine the surcharge on the gold, except in the mint, where one proof centre is run in the middle of a muffle full of about 16 assays, but ¼ point to 2 points is subtracted from the gold according to the judgment of the assayer, based on his previous experience with the method he uses. From 3 to 15 points may also be added to the silver to correct for cupellation loss, but this is much larger and much less constant than surcharge, so that it is well to use a proof centre made up of parted gold, test silver and copper, unless the assayer is constantly doing such work, in case silver must be accurately determined.

Hence to obtain the corrected gold fineness, subtract the surcharge from the weight of the parted gold; to obtain the corrected silver fineness, add together the surcharge, the cupellation

loss and the uncorrected silver fineness; and to obtain the corrected base metal, subtract the cupellation loss from the uncorrected base metal.

The test silver should be at least 0.998 in silver as purchased and should contain no gold or at least less than 0.10 mg. in 1 grm.

The proof gold which you are using was prepared by the method of Robert-Austen, as follows: "The purest gold obtainable (cornets from previous bullion assays) are dissolved in *aqua regia*, evaporated to drive off excess of acid, diluted with distilled water so that the solution does not obtain more than one-half ounce of gold per gallon, allowed to stand several days and the gold solution poured off from the silver chloride through a filter. The gold is then precipitated with oxalic or sulphurous acid, washed with distilled water, then with hydrochloric acid, ammonia and water in succession to remove traces of silver chloride, dried, melted in an earthern crucible with borax, cleaned with hot, strong sulphuric aid, washed, dried and rolled out." As stated above, it is not generally customary nor is it necessary to prepare this pure gold for proof centres; we do it here that you may have an absolute check on the method in this, your first assay of gold bullion, and on the sources of error in all gold assays.

Read *before* beginning the assay:

1. Furman's "Assaying," pp. 136-137 and p. 308.

2. The Fire Assay of Lead Ores. J. F. Cannon. Eng. and Min. Journal. Copy this article *verbatim* into your notes.

Prepare one charge of each of the following ores and make the fusion in the muffle, using 10-gram crucibles.

1. Sample 22. Pure assayer's litharge, dried. Sample 10 grams. Sodium bi-carbonate 12 gms. Potassium carbonate 12 gms. Argol 1½ gms. Mix. Cover with salt.

2. Sample 35. Pure galena. Sample 10 gms. Sodium bi-carbonate 12 gms. Potassium carbonate 12 gms. Argol 3 gms. Mix. Add 5 gms. of ground borax on top, stick two or three 12-penny nails into the charge and cover with salt.

3. Sample . A high grade galena concentrate. Use the same charge as for 2.

Potassium carbonate is very deliquescent and no particle of it should be left on the scale pan or the table; and the can containing it or the lead flux ought not to be left uncovered.

The above three charges are to be boiled down at a moderate red, so that it will be about twenty minutes from the time they are put in until they are boiled down, it being supposed that they will begin to boil in from five to eight minutes after putting in. When boiled down, charge 1 is to be given a low yellow heat for five minutes, taken out and poured. Now fill the mouth of the muffle with coke and give charges 2 and 3, since they are sulphide ores, a bright yellow heat for about 15 minutes, until the nails can be removed free from lead, as in the nail assay for gold. Pour into a scorification mould, hammer off the slag and clean with a brush; or with dilute hydrochloric acid, dry and weigh to .01 gm. Report lead to 1-10%.

CHEMISTRY OF LEAD ASSAYS.

The chemical reactions which go on in the reduction of galena to lead by iron and an alkaline flux have been given as:

(1.) $7PbS + 4Na_2CO_3 = 3Na_2PbS_2 + Na_2SO_4 + 4CO_2 + 4Pb$

(2.) $3Na_2PbS_2 + 3Fe = 3Na_2S + 3FeS + 3Pb.$

The first reaction goes on at a moderate red and has taken place when the boiling is finished; for the second re-action, a very high heat, continued for some time, is needed. Galena is

volatile by itself, as may easily be proved by heating some in a glass tube closed at one end, and metallic lead, also, is readily volatile. The heat should, therefore, be low during the boiling to reduce the loss of lead by its volatization as sulphide or as metallic lead to as small an amount as possible: it cannot be entirely prevented; the heat must be very high at the end, that the iron may leave as little lead as possible unreduced in the slag: there will always be a small slag loss. There is little danger of volatilization after the boiling is over, since the lead is collected in a button at the bottom of the crucible and is covered with a thick layer of strongly reducing slag.

It is the universal experience of lead assayers that the best results are obtained by managing the heats in this way; beginners generally fail at first through getting the assays too hot when boiling and not hot enough or for a sufficient time at the final heat.

Make up some lead flux by making a thorough mixture of: sodium bi-carbonate one pound, potassium carbonate (sal tartar) one pound, argol ¼ lb. One measure even full contains: 12 grams $NaHCO_3$, 12 grams K_2CO_3, 3 grams of argol, and, with borax, nails and a salt cover, will flux 10 grams of *any* lead ore. The charge melts easily, is of universal application and gives, I have found, the highest results attainable by any method (except a cyanide flux) when the fusion is made in the muffle; and it is commonly used at smelting works in this country and in Mexico.

Also make the lead flux described by Cannon in the article which you have copied.

Fusion in the muffle is given the preference, as then the heat can be evenly applied and easily controlled. Sodium carbonate can be used in place of the potassium salt when necessary, somewhat more being needed: the two are used together to make a more readily fusible flux

Make assays of a rich sulphide ore using both fluxes and compare results; and use one or the other on each of the samples which follow. Pour some onto an iron plate (the buck-board), as described by Cannon.

4. Sample . Carbonate lead ore; consists largely of cerussite. Use one or both lead fluxes and assay the sample in duplicate. One nail is always added to oxidized ores, whether they contain small amounts of sulphides or not, because some anglesite (lead sulphate) may be present. Use only 5 grams of

ground borax on top.

5. Sample . A low grade lead ore containing large amounts of other metallic sulphides (pyrite, chalcopyrite, zinc blende). Use one or both lead fluxes, three 12-penny nails and make the assay in duplicate. Use 10 gms. of ground borax on top.

6. Sample 342. Tailings from concentrating a carbonate lead ore. Run duplicates, each; Tailings 25 gms., lead flux 65 gms. Salt Cover. 1 nail. Pour into a mould.

For methods of assaying lead slags, see Furman, p 308, and Hofman's "Metallurgy of Lead" pp 70 and 71.

No payment is generally made by the smelters for lead in ores when the content is under 5%.

The use of potassium cyanide (30 gms. of potassium cyanide to 10 gms. of a carbonate ore or 30 gms. of potassium cyanide and 5 gms. of potassium ferrocyanide for an ore containing sulphides) as a flux for lead ores has been proposed. On pure, rich ores, it gives results ½% to 1% higher than the best nail assay; it is totally inapplicable to low grade ores or ores containing other reducible metals. It is never accepted as the basis for the purchase of lead ores nor is it so reliable as the nail assay, because other base metals are more apt to be reduced along with the lead than by the nail assay and hence it is not of universal application. For these reasons it is not worth while to practice at it.

An excess of reducing agent is always added to the charge in making lead assays above the amount necessary to reduce 10 grams of lead oxide; but if too large an excess be added, the slag will be rendered less readily fusible by the separated carbon suspended in it. Charcoal or flour can be substituted for argol, but the latter is to be preferred as a reducer in lead assays, since the carbon separates from it in a very finely divided state on heating and the potassium carbonate formed by its decomposition assists in forming a fluid slag.

Some consider that the borax is better added on top of the charge, since if lead oxide is once slagged by the borax the lead is apt not to be as completely reduced. Some borax is generally necessary to flux the gangue of the ore and render fluid slags containing much iron matte; but no large excess of it should be used.

Compare the results by volumetric wet assay with those by fire assay on any samples on which you use both methods.

ADDITIONAL REFERENCES ON LEAD ASSAYS.

1. The Fire Assay for Lead. Malvern W. Iles. School of Mines Quarterly, Vol. 15, p. 336. Shows in detail the errors made in fire assays for lead and gives results by fire and wet assays on many samples, whose analyses are given. Should be read by all.

2. Aaron's "Assaying," Part III, pp. 94-96.

3. Percy's "Metallurgy of Lead," pp. 103-120.

4. Bodemann & Kerl's "Assaying," pp. 17-48. Gives a good account of the chemistry of the lead assay, although it describes methods which have been superceded by better ones.

5. Hofman's "Metallurgy of Lead," pp. 70-71.

ASSIGNMENT 22. FIRE ASSAY OF IMPURE LEAD ORES.

The errors inherent in all fire methods for assaying lead ores may be grouped under three heads:

1. Lead is lost by the volatilization of the metal or its compounds.
2. Lead is lost by passing into the slag.
3. Other reducible metals are precipitated with the lead and are weighed and reported as lead.

The only base metals commonly occuring in lead ores which cause serious errors by contaminating the lead button are copper and antimony. When malachite or any antimony mineral is present in considerable amount in a lead ore, the fire assay no longer yields even fairly satisfactory results and a wet determination must be made.

EFFECT OF COPPER.

1. Mix together 5 gms. of litharge and 5 gms. of copper carbonate and fuse with the fluxes given in the last assignment for the carbonate ore. Compare the percentage of lead given by the button with the percentage known to be present

2. Repeat, the same in all respects, but use 3 gms. of sulphur in addition to the other fluxes.

3. Mix 5 gms. of copper matte with 5 gms. of pure galena and use the lead flux. Make the same calculations as above.

Test each of the three buttons for copper as follows: dissolve 1 gm. of the clean button in a small amount of dilute nitric acid, precipitate the lead with sulphuric acid and filter. Make the filtrate alkaline with ammonia. Titrate the blue solution for copper.

The first charge will produce a coppery button and is given to show the effect of oxidized copper minerals in an ore. In the second, the sulphur is used to convert tne copper carbonate into copper sulphide, as far as possible, and so retain it in the slag, in the form of matte.

EFFECT OF ARSENIC.

Mix 5 gms. of pure galena with 5 gms. of arsenopyrite, make a regular lead assay, separate and weigh both the lead and the speiss, calculate the percentage of lead from the button and compare with the true percentage. Test the hardness, tenacity and malleability of both lead and speiss with the hammer and anvil, and compare with pure lead. Roast some of the speiss on charcoal and notice the odor of the arsenic fumes.

EFFECT OF ANTIMONY.

Repeat the last experiment, substituting 5 gms. of stibnite (sulphide of antimony) for the arsenopyrite. Make the same calculations, test the button with hammer and anvil as before and test 1 gm. of the button for antimony as follows: Treat with hot 1.15 nitric acid until all acted on, pour off the solution of lead nitrate, wash the white precipitate and test it for antimony by fusing on charcoal before the reducing flame of the blowpipe with twice its bulk of soda.

Ores containing large amounts of antimony must have their lead determined by the wet assay.

The following statements on "The Influence of other Metals on the Lead Assay" are copied from John Percy's treatise on "The Metallurgy of Lead," a book which is out of print and rare:—

"*Silver.* During the assay, practically the whole of the silver passes into the lead; but the quantity is generally insufficient to add materially to the quantity of lead obtained. Moreover, supposing it to be present to a greater extent, it is customary to report the lead as containing silver, and therefore, the total weight of lead obtained by assay would be reported inclusive of the silver present.

"*Gold.* This metal is always present in lead ores but generally in unweighable quantity in the amount of ore operated on; and it may be practically disregarded.

"*Copper.* This metal is sometimes present in lead ores in the state of copper pyrites, chalcocite or carbonate of copper.

During the assay, part of the copper passes into the lead and part remains in the slag, the quantity which passes into the lead varying according to the nature of the associated cupriferous minerals and the nature and proportions of the reducing agents employed. (Little copper is reduced into the lead when it is present as chalcopyrite and there is much pyrite in the ore; but a large amount is reduced when the copper occurs as carbonate and the ore contains little sulphur.)

"*Zinc.* This metal is sometimes present in lead ores in the state of blende or calamine. During assaying, part of the zinc is volatilized and part remains in the slag, and only a very small portion (a trace) passes into the lead.

"*Antimony.* This metal is generally present in lead ores in the state of sulphide. During assaying, a large proportion of antimony passes into the lead. The buttons of lead are harder and whiter than those obtained from ordinary galena; their fracture is bright and granular. (I have found that a considerable proportion of antimony is sometimes found with iron in a separate speiss button.)

"*Arsenic.* This metal is sometimes found present in lead ores in the state of arseniate of lead or of arsenical iron pyrites. During assaying, part of the arsenic is volatilized in the state of arsenic or sulphide or arsenic and part enters into combination with iron and separates on cooling on top of the lead as speiss, or arsenide of iron, of the formula Fe_6As, which may be detached from the lead by hammering. The lead is practically free from arsenic.

"*Iron.* This metal is sometimes present in lead ores in the state of iron pyrites. During assaying, it passes into the slag, forming a double sulphide of iron and sodium, and does not exert any marked influence upon the yield of lead."

From the above it is evident that the only elements liable to produce errors by contamination of the lead button with foreign substances are copper and antimony. Percy gives results of experiments which he made and which support this conclusion.

Suggestions on Fitting up an Assaying Laboratory.

It is intended to offer here a few suggestions on fitting up an assaying laboratory under the conditions ordinarily found at mines, choosing the tools and apparatus with the idea of securing an efficient equipment at the least expense.

The chief part (perhaps two-thirds) of the expense will be in the purchase of the balances and the furnaces. Some tools can be improvised but the bead balance, at least, must be a good one as purchased. A proper balance, even for a small mine, cannot be bought for much less than one hundred dollars; any bead balance of a lower grade is not a good investment. Many of the larger mines have a fine balance of Oertling's or Ainsworth's make. Where only occasional assays are to be made and economy is necessary, Becker's 1A balnace, costing about sixty dollars, will answer if solidly set, adjusted sensibly, and carefully used. Refer to assignment 1.

The furnace is next in importance after the bead balance. Where the choice lies between charcoal and gasoline, choose the latter for economy, cleanliness and convenience. If coke is burned, the stack should be at least 15 feet high with a flue 5 inches in diameter; charcoal does not require nearly so strong a draft as coke. In a place very hot in summer, the drum of gasoline is best kept in a drift run five to ten feet into any sloping ground near the assay office.

A drum of gasoline contains 110 gallons and costs $15.00 to $16.00 in San Francisco; the gasoline weighs about 700 lbs. and the drum about 250 lbs. A drum full of gasoline will run one burner about 325 to 350 hours, or say four hours each day for three months. The cost is about five cents per hour, with gasoline at this price.

Although all the operations of the laboratory can be and very commonly are all done in the same room, there should preferably be one room for sampling and furnace work and with a sink for panning, another for parting and for chemical work and a third to contain the bead balance and a writing shelf. The furnaces may well occupy the darkest part of the laboratory; the parting shelf should be just at a window and facing it. The bead balance should be so placed that the direct rays of the sun cannot shine on it and so that the weigher gets the light over his left shoulder. The bead balances and the mortar should each be placed on a post going into the ground and independent of the floor. The pulp scale should be placed on a broad plain

table say six feet long by three feet wide, on which stand a dust-tight flux box divided into compartments, with hinged lid.

Sampling. Provide 25, 50 or 100 duck sample bags, twelve inches by six inches, or larger, for bringing samples from the mine to the laboratory, and number them consecutively with a stencil. The best hand tools are a rotary hand crusher, such as the Bosworth, or a large mortar with heavy pestle for breaking to about 10 mesh and a flat plate 20x24 inches with low rims at the sides, with a 10 lb. and also a 40 lb. muller for fine grinding. See that the meshes of the number 100 screen bought are evenly spaced. A small sample grinder or jaw crusher, placed in the mill and driven from the shafting, although seldom provided, is a great aid in making thorough samples of ore from the mine. Use sample pans and paper envelopes for holding the assay pulps.

Parting. As gas is not available, other sources of heat must be used. A convenient arrangement is to use a small kerosene stove to heat a sand-bath (a granite iron pie-plate filled with tailings) or a piece of heavy sheet iron on which the capsules are placed; the annealing is done over an alcohol lamp with a flame of good size, thin capsules being chosen that the heat may readily penetrate them; or the gold is annealed by putting the capsules into a tray or crate of sheet iron or copper of a size to go readily into the muffle and perforated with holes to fit the capsules and the whole is put into the muffle, a long handle being riveted to the tray so that no tongs are necessary. Another method is to remove one side of a box of suitable size and replace the top with a sheet of card asbestos perforated with a dozen or two holes to receive the capsules; the alcohol lamp is then moved around under them with the hand to keep the acid at the proper heat during parting and the annealing is done in the same apparatus. A third way is to obtain a hot plate by putting a heavy piece of sheet iron on top of the round furnace or gasoline crucible furnace; but the heat of the plate is not readily controlled. Either the first or second method is better than the third.

Laboratory Records:—There should be kept in the laboratory a book of convenient size to contain complete records of all assays made. It is most convenient to keep it in hard pencil as it contains the weights as taken down at the balance. Some select a book four inches by six inches bound in leather like a transit book and use a separate page for the record of each assay. Others select a large, well bound book with ruling like an

account book and leave a line or two between the record of each assay. The recording of all weights and results as obtained in such a book not only prevents confusion and the losing of the results of assays but the record may be needed to refer to for some purpose in working the mine several months or years later.

The record of each assay should show date, number of sample (consecutive numbers being used) description or mark (tails, concentrates, mine sample, with point in mine where each sample was taken,) ore used in assay tons, weights of dore, Au, Ag, Oz. per ton of Au aud Ag, and value per ton in gold. By having a definite place on the page for each item, such words as A. T., dore, sample number, oz. per ton, etc. can be omitted. Of course this book is the property of the mine, not of the assayer.

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